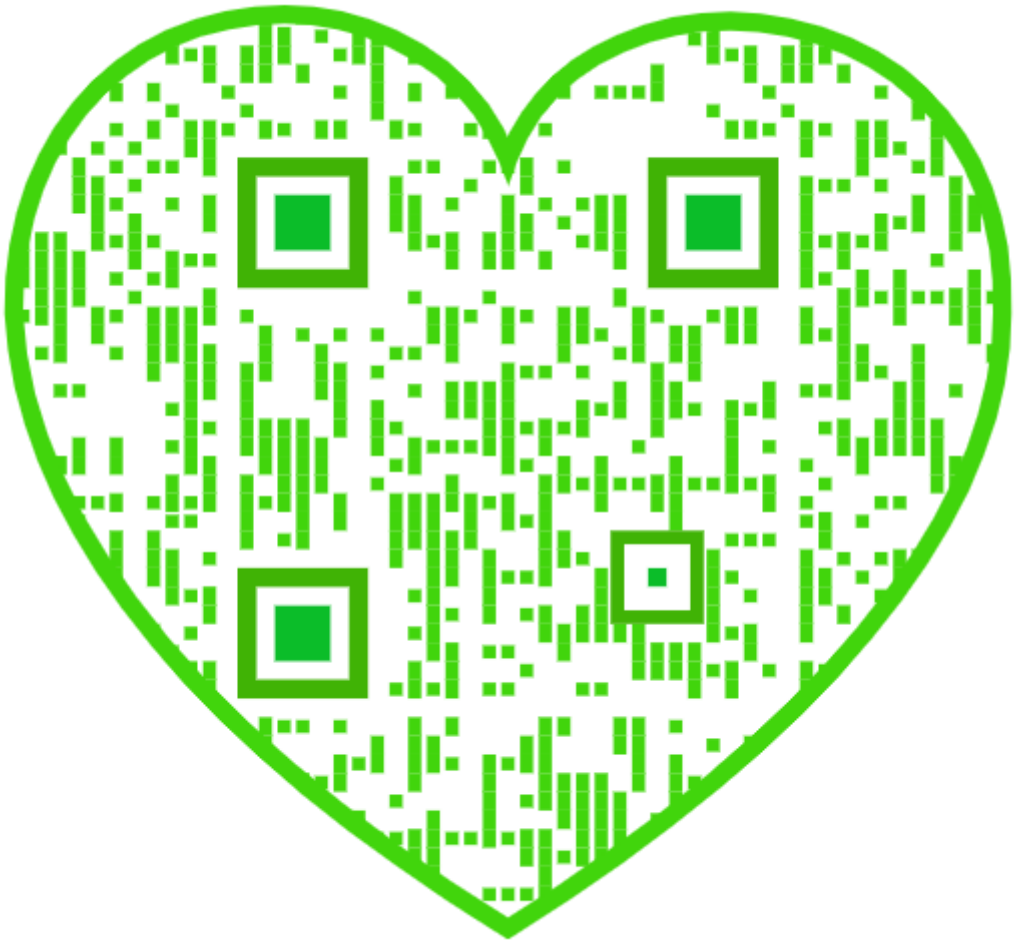

INSTRUCTIONS

FOR THE

ANALYSIS OF SOILS, LIMESTONES,

AND

MANURES.

BY

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PREFACE TO THE THIRD ENGLISH EDITION.

This little work is not intended to compete with treatises on chemical analysis, such as those of Rose and Fresenius, which are the text-books of the accomplished analyst. Originally published as an Appendix to my *Lectures on Agricultural Chemistry and Geology*, it has been much in demand also in a separate form. I have, therefore, given to this third edition a more widely practical bearing, by including *limestones, clays, ironstones, manures,* and *natural waters,* among the substances to be analysed. I have briefly explained, also, the principles on which analysis by measure is founded—a method which is susceptible of many simple practical applications. The Instructions are as few and simple as the subject well admits of, and the advancing student will proceed from this little manual to the many larger works which are within his reach.

To the schoolmaster, the farmer, the pharmaceutical chemist and druggist, the youthful student, and to the rural, the training or normal school, and the agricultural laboratory, I offer it as a FIRST HELP TO PRACTICAL AND ECONOMICAL CHEMICAL ANALYSIS. Though small in size, it will materially aid them in those chemical investigations which, in connection with agriculture and the arts, are every day becoming more sought for, and more necessary.

With a view to easy reference, I have added a copious Index.

DURHAM, March 1855.

AMERICAN PUBLISHER'S PREFACE.

THE want in this country of just such a work as Prof. Johnston has here given to the world, there being nothing of the kind in the United States, has induced the publisher to issue an edition of it for the benefit of the farmers of America, in a cheap form. The importance to the Agriculturist of a thorough knowledge of all branches of his business, and of the fundamental principles which govern the development of vegetation, and its perfect growth, need not be dwelt upon. Every man who understands his own interests, desires a knowledge of everything which will promote them, and I have not a remote doubt that a careful study of this little book, aided by experiments in accordance with its instructions, will redound in large benefits to the student. Few and simple as the Instructions are, they yet form one of the most valuable aids to the science of Agriculture, which have ever been published.

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INSTRUCTIONS

FOR THE

ANALYSIS OF SOILS, LIMESTONES, MANURES, &c.

CHAPTER I.

PHYSICAL PROPERTIES OF THE SOIL.

Why a soil should be analysed.—Usefulness of knowing the proportions of lime, organic matter, and sand or clay in a soil.—More refined inquiries.—How to select a soil for examination.—To determine the physical properties of a soil; its density, absolute weight, proportions of sand and gravel; its absorbing power; its power of retaining water; rapidity with which it dries; power of absorbing heat from the sun; and rapidity of cooling.—The sandy deserts.

§ I. WHY A SOIL SHOULD BE ANALYSED.

1°. The benefits to be derived from the chemical examination and analysis of a soil are by many misunderstood. Some have represented it as the only sure guide to successful cultivation; while others have not scrupled to pronounce the analysis of soils to be entirely useless, and unfitted to lead to any profitable practical result. Both of these extreme parties are in error. For while it is often very difficult, from an analysis alone, to explain either the

past agricultural history, the present money value, or how best to remedy the known defects of a soil, yet there are many practical points on which analysis does throw light, and modes of practical treatment which it serves at once either to discourage or to recommend.

2°. Thus, on many accounts, it is desirable to know how much lime a soil contains. Soils rich in lime generally produce a sweet herbage, sound and nutritious green crops, and grain of a full ear and strong straw. To secure these advantages, the farmer is willing to apply lime; but the land may contain enough already, and to apply more might only be a waste; or it may contain little or none, and he may be about to apply too little. A simple analysis settles difficulties of this kind, determines the per-centage of lime, and points out what in the circumstances is best to be done.

3°. Again, the proportion of organic or combustible matter in a soil regulates, in some degree, the dose of lime it is proper to add—the kind of mineral, vegetable, or other manure it may be proper to use upon it—and in the case of moorish or peaty soils, how far clay or sand would be likely to improve it. Hence it is desirable to know what per-centage of organic matter a soil contains.

4°. Again, a poor soil has sometimes much resemblance to clay, and yet, on chemical examination, proves to consist mainly of a very fine sand. To ascertain this, is to determine at once how the soil may be improved. Or a soil may be found, upon analysis, to be too largely impregnated with oxide of iron, or to contain peculiarly noxious combinations of iron, or to be too rich in common salt; and each of these results of analysis indicates to the

skilful man the steps which will most quickly or economically bring the several soils into a fertile condition.

5°. These are some of the plainer cases in which useful information may be derived from comparatively simple analyses of soils. When the skill of the analyst is sufficiently refined to enable him to detect or determine the phosphoric acid, the potash, the ammonia, or the nitric acid which a soil contains, other conclusions may be drawn which are not without their practical value. It is the pretended application of such determinations—made hastily and without sufficient care or knowledge, either chemical or agricultural—which have led many to deny as hastily, that any good at all is to be derived from the analysis of a soil.—(See Chap. VII.)

§ II.—HOW TO SELECT SPECIMENS OF SOILS FOR ANALYSIS.

6°. In the same field, different varieties of soil often occur; and some recommend that, in collecting a specimen for analysis, portions should be taken from different parts of the field and mixed together, by which an average quality of soil would be obtained. But this is bad advice, when the soils in different parts of the field are really unlike. Suppose one part of a field to be clay, and another sandy—as is often the case in most countries—and that an average mixture of the two varieties of soil is submitted to analysis, the result obtained will apply neither to the one part of the field nor to the other; that is, it will be of little or no practical value. In selecting a specimen of soil, therefore, one or two pounds should be taken from each of four or five parts of the fields where the soil

appears nearly alike. These should be well mixed together, and dried in the open air, or before the fire. Two separate pounds should then be taken from the whole, for the purpose of analysis; or, if it is to be sent to a distance, should be tied up in clean strong paper; or, what is much better, should be enclosed in clean, well-corked bottles.

§ III.—TO DETERMINE THE PHYSICAL PROPERTIES OF THE SOIL.

7°. *Determination of the density of the soil, or its weight compared with that of water.*—In order to determine the density of the soil—or its specific gravity, as it is also called—a portion of it must be dried at the temperature of boiling water (212° Fahr.), till it ceases to lose weight; or upon a piece of white paper, in an oven, at a heat not great enough to render the paper brown. A common phial or other small bottle, perfectly clean and dry, may then be taken and filled with distilled or pure rain-water, up to a mark made with a file on the neck, and then carefully weighed. Part of the water may then be poured out of the bottle, and 1000 grains of the dry soil introduced in its stead. The bottle must then be well shaken, to allow the air to escape from the pores of the soil;—filled up again with water to the mark on the neck, and again weighed. The temperature of the water should all the while be kept as near to 60° Fahr. as possible. The weight of the soil—divided by the difference between the weight of the bottle when it contains the soil and water, and the sum of the weights of the soil and of the bottle of water added together—gives the specific gravity.

Thus, let the bottle with water weigh 2000 grains, and with water and soil 2600, then—

	Grains.
The weight of the bottle with water alone =	2000
The weight of the dry soil,	1000
Sum—being the weight which the bottle with the soil and water would have had, could the soil have been introduced without displacing any of the water,	3000
But the weight of the bottle with soil and water was	2600
Difference—being the weight of water taken out to admit 1000 grains of dry soil,	400

Therefore 1000 grains of soil have the same *bulk* as 400 grains of water; or the soil is $2\frac{1}{2}$ times heavier than water, since $\frac{1000}{400} = 2.5$ its specific gravity.

8°. *Determination of the absolute weight.*—The absolute weight of a cubic foot of solid rock is obtained in pounds by multiplying its density or specific gravity by $63\frac{1}{2}$—the weight in pounds of a cubic foot of water. But soils are porous, and contain more or less air in their interstices, according as their particles are more or less fine, or as they contain more or less sand or vegetable matter. They are not so heavy, therefore, as the solid rocks from which the are formed. Fine sands are the heaviest, clays the next in order, and peaty soils the lightest. The simplest mode of determining the absolute weight of a soil is to weigh an exact imperial half-pint of the soil in any state of dryness, when this weight, multiplied by 150, will give very nearly the weight of a cubic foot of the soil in that state.

9°. *Determination of the relative proportions of gravel, sand, and clay.*—Five hundred grains of the dry soil may be boiled in a flask, or in a small enamelled iron pan, half full of water, till the particles are thoroughly separated

from each other. Being allowed to stand for a couple of minutes, the water, with the fine matter floating in it may be poured off into another vessel. This may be repeated several times, till it appears that nothing but sand or gravel remains. This sand and gravel is then to be washed completely out of the flask or pan, dried, and weighed. Suppose the weight to be 300 grains, then 60 per cent * of the soil is sand and gravel. The sand and gravel are now to be sifted through a gauze sieve more or less fine, when the gravel and coarse sand are separated, and may be weighed and their several proportions estimated.

These separate portions of gravel and sand should now be moistened with water and examined carefully with the aid of a microscope, with the view of ascertaining if they are wholly silicious, or if they contain also fragments of different kinds of rock—sand-stones, slates, granites, traps, lime-stones, or iron-stones. A few drops of strong muriatic acid (spirit of salt) should also be added. The presence of lime-stone is then shown more distinctly by an effervescence, which can be readily perceived by the aid of the glass—of peroxide of iron, by the brown colour which the acid speedily assumes—and of black oxide of manganese, by a distinct smell of chlorine, which is easily recognised. In the subsequent description of the soil, these points should be carefully noted.

Suppose the sand and gravel to contain half its weight of fine sand, then our soil would consist of—coarse sand and small stones 30 per cent, fine sand 30 per cent, clay and other lighter matters 40 per cent. When the sand is

* As 500 : 300 : : 100 to 60.

very fine, care must be taken that none of it is washed off and reckoned as clay.

10°. *Absorbing power of the soil.*—A thousand grains of the soil, made perfectly dry, as described in 7°, should be crushed to powder, spread over a sheet of paper, exposed in the open air in ordinary dry weather, shaded from the sun for twelve or twenty-four hours, and then weighed. The increase of weight shows its power of absorbing moisture from the air. If it amount to 15 or 20 grains, it is so far an indication of great agricultural capabilities.

11°. *Its power of holding or retaining water.*—This same portion of soil may next be put in a funnel upon a *double** filter, and cold water poured upon it, drop by drop, till the whole is wet and the water begins to trickle down the neck of the filter. It may now be covered with a piece of glass, and allowed to stand for a few hours, occasionally adding a few drops of water, until there remains no doubt of the whole soil being perfectly soaked. The two filters and the soil are then to be removed from the funnel, and the filters opened and spread for a few minutes upon a linen cloth, to remove the drops of water which adhere to the paper. The wet soil and inner filter being now put into one scale, and the outer filter into the other, and the whole carefully balanced, the true weight of the wet soil is obtained. Suppose the original thousand grains now to weigh 1400, then the soil is capable of holding 40 per cent of water.†

12°. *Rapidity with which the soil dries.*—The wet soil

* That is one filter within another.

† 1000 : 400 the increase of weight as 100 : 40.

with its filter may now be spread out upon a plate, and exposed to the air, in what may be considered the ordinary circumstances of temperature and moisture of the place, for four, twelve, or twenty-four hours, and the loss of weight then ascertained. This will indicate the comparative rapidity with which such a soil would dry, and the consequent urgent demand for draining, or the contrary. As great a proportion of the water is said to evaporate from a given weight of silicious sand saturated with water, in four hours, as from an equal weight of pure clay in eleven, and of peat in seventeen hours—when these several soils are all placed in the same circumstances.

In making this experiment, a portion of pure quartz sand or of pipe-clay may be employed for the purpose of obtaining a *comparative* result as to the rapidity of drying.

13°. *Power of absorbing heat from the sun.*—The same method may be adopted in regard to the power of the soil to become warm under the influence of the sun's rays. Two small wooden boxes, containing each a layer, two or three inches in depth, of one of the kinds of soil which are to be compared, may be exposed to the same sunshine for the same length of time, and the heat they severally acquire determined by a thermometer, the bulb of which is buried a full quarter of an inch beneath the surface. Soils are not found to differ so much in the actual temperature or degree of warmth they are capable of attaining under such circumstances—most soils, when dry, becoming 20° or 30° warmer than the surrounding air in the time of summer—as in the relative *degree of rapidity* with which they acquire this maximum temperature.

This rapidity depends very much upon the darkness of the colour of the soil. When the proportion of organic matter is great, the soil is generally very dark in colour, and the absorption of heat from the sun most rapid.

But the mineral constituents of the soil also influence this quality. Of all the known constituents of soils, dry quartz sand absorbs heat most rapidly. It reaches the maximum temperature, on a sunny day, nearly five times sooner (as 90 to 19—TINDALL) than a similar surface of dry gypsum. To this circumstance is to be ascribed both the pleasant warmth of the sand-hills along our sea coasts on a summer day, and the scorching heats which distinguish both air and soil in regions of sandy desert.

14°. *Rapidity of cooling.*—The above property derives another practical interest from its being connected with a corresponding rapidity of cooling. Dark soils cool most rapidly, and mists and fogs settle over them, and dews fall and moisten them. So, also, naked sandy soils and plains part with their heat as rapidly as they take it in. Hence the chill nights which succeed the fiery noondays of the African Sahara---and hence the alternate heats and chills which burn up thin sandy pastures, and make dark, moorish, or peaty lands unpropitious to our cultivated crops.

CHAPTER II.

ORGANIC MATTER OF THE SOIL.

Determination of the organic matter of the soil; the proportion or per-centage. The humic and ulmic acids.—The insoluble vegetable matter, or humus. Other organic substances in the soil; how determined.

15°. *Determination of the per-centage of organic matter.* The soil must be thoroughly dried in an over or otherwise—in a little sand-bath for example, over a lamp or charcoal fire—at a temperature not higher than from 250° to 300° Fahr. The humic and ulmic acids of soils* will bear the latter temperature without change. An accurately weighed portion (100 to 200 grains) must then be burned in the open air, till all the blackness disappears. This is best done in a small platinum capsule, over an argand, spirit, or gas lamp. The loss indicates the total weight of organic matter present. It is scarcely ever possible, however, to render soils absolutely *dry*, without raising them to a temperature so high as to char the organic matter present. Hence the weight of the organic matter, as determined from the loss, will always somewhat *exceed* the truth; the water which still

* See the author's *Lectures on Agricultural Chemistry and Geology*, 2d edition, p. 71; or his *Elements*, 6th edition, p. 21.

remains in the soil being driven off along with the organic matter, when the soil is heated to redness. This excess, also, will in general be greater in proportion to the quantity of clay in the soil, since this is the ingredient of most soils from which the water is expelled with the greatest difficulty.—(See under 18° *c.*)

16°. *Determination of the humic and ulmic acids.*—These acids, whether merely mixed with the soil, or combined with some of the lime and alumina it contains, are extracted by boiling with a solution of the common crystalised soda of the shops. Into about two ounces by measure of a saturated solution of this soda, contained in a flask, 200 or 300 grains of soil, previously reduced to coarse powder, are introduced, an equal bulk of water added, and the whole boiled or digested on the sand-bath, with occasional shaking for an hour or two. The flask is then removed from the fire, filled up with water, well shaken, again boiled, and the particles of soil afterwards allowed to subside. The clear liquid is then poured off. If it has a brown colour, it has taken up some humic or ulmic acid. In this case, the process must be repeated once or twice with fresh portions of the soda solution, till the soluble organic matter appears, by the pale colour of the last solution to be altogether taken up. These coloured solutions are then to be mixed and filtered. This filtering generally occupies considerable time, the humic and ulmic acids clogging up the pores of the filter in a remarkable manner, and permitting the liquid to pass through sometimes with extreme slowness.

When filtered, muriatic acid is to be slowly added to the coloured liquid—which should be kept in motion by

a glass rod—till effervescence ceases, and the whole has become distinctly sour. On being set aside, the mixed ulmic and humic acids fall in brown flocks. A filter is now to be dried and carefully weighed,* the liquid filtered through it, and the acids thus collected. They must be washed on the filter with pure water—rendered *very* slightly sour by muriatic acid†—till all the soda is separated.‡ The filter with its contents is then to be dried at 250° Fahr., till it ceases to lose weight. The final weight, when the known weight of the filter is deducted from it, gives the quantity of humic and ulmic acids contained in the portion of soil submitted to examination. As it is rarely possible to wash these acids perfectly upon the filter, rigorous accuracy requires that the filter and acids should be burned after being weighed, and the weight of ash left, minus the known weight of ash left by the filter,§ deducted from that of the acids

* This is best effected by putting the filter into a small covered porcelain crucible of known weight, and heating it for ten minutes over a lamp or otherwise, at a temperature which just does not discolour the paper, allowing then the crucible to cool under cover, and when cold weighing it. The increase above the known weight of the crucible is that of the filter, which, besides being recorded in the experiment-book, should also be marked in several places on the edge of the filter with a black-lead pencil.

† This is to prevent the humic acid from passing through the filter, which it is very apt to do, when the saline matter is nearly washed out of it.

‡ This is ascertained by collecting a few drops of what is passing through upon a slip of clean glass or of platinum foil, and drying them over the lamp, when, if a perceptible stain or spot is left, the substance is not sufficiently washed.

§ The ash left by the paper employed for filters should always be known. This is ascertained, once for all, by drying a quantity of it in the way described in the previous note, weighing it in this dry state, burning it, and again weighing the ash that is left. In good filtering paper, the ash ought not to exceed one per cent.

as previously determined. It is to be observed here that by this, which is really the only available method we possess of estimating the humic and ulmic acids, a certain amount of loss arises from their not being wholly insoluble, the acid liquid which passes through the filter being always more or less of a brown colour.*

The two acids thus estimated occur in soils mixed together in various undetermined proportions. It is not easy to separate them from each other, and no known practical good would result from a rigorous determination of the proportions of each.

17°. *Determination of the insoluble vegetable matter or humus.*—Many soils, after this treatment with carbonate of soda, are still more or less of a brown colour, evidently due to the presence of other organic matter. To separate this, it is recommended to boil the soil, which has been treated with carbonate of soda, and which we suppose still to remain in the flask, with a solution of caustic potash, repeated, if necessary, as in the case of the soda solution. By this boiling, the vegetable matter, which was insoluble in the carbonate of soda is changed in chemical constitution, and dissolves in the caustic potash, giving a brown solution. From this it may be separated in brown flocks by the addition of muriatic acid, and then collected and weighed as above described.

In some soils, also, distinct portions of scarcely altered

*The portion which thus remains in solution may be precipitated by adding a small quantity of a solution of alum, and afterwards pouring in ammonia in excess. The alumina falls coloured by the organic matter, and after being collected on a filter, washed and dried, the weight of organic matter in the precipitate may be determined approximately as described under 15°.

vegetable fibre—such as portions of roots, &c.—are present, and may be separated, mechanically dried, and weighed.

18°. *Of other organic substances present in the soil.*—The sum of the weights of the above substances, deducted from the whole weight of organic matter, as determined by burning, gives that of the *other* organic substances present in the soil. The quantity of these is in general comparatively small; and unless they are soluble in water, there is no easy method of separating them and determining their weight. The following two methods, however, may be resorted to:—

a. Half a pound or more of the moist soil may be boiled with two separate pints of distilled water, the liquid filtered and evaporated to a small bulk. From clay soils, when thus boiled with water, the fine particles do not readily subside. Sometimes, after standing for several days, the water is still muddy, and passes muddy through the filter; but after being evaporated, as above recommended, to a small bulk, most of the fine clayey matter remains on the paper when it is again filtered. As soon as it has thus passed through clear, the liquid may be evaporated to perfect dryness at 250° Fahr. and weighed. Being now treated with water—a portion will be dissolved—this must be poured off, and the insoluble remainder again perfectly dried at 250° Fahr., and weighed. If this remainder be now heated to redness in the air, any organic matter it contains will be burned off, and the weight of this organic matter will be indicated by the loss on again weighing. This loss may be considered as humic, ulmic, or some other acid

rendered insoluble by the drying at 250.* It does not require to be added to the weight of humic acid already determined (16°) because in that experiment a portion of soil was employed which had *not been boiled in water*, and from which, therefore, the carbonate of soda would not at once extract *all* the humic and other acids. The present experiment need only be made when it is desirable to ascertain what weight of acid organic matter a soil contains in a state in which it is soluble in water. Where carbonate of ammonia, of potash, or of soda is present in the soil, this quantity may be very considerable, and may exercise an important influence upon vegetation.

b. That which was taken up by water from the dried residuum is again to be evaporated to dryness dried at 212°, weighed, and burned at a *low* red heat. The loss is organic matter, and may have been crenic or apocrenic, or some other of the organic acids which are formed in soils, and the compounds of which, with lime, alumina, and protoxide of iron, are soluble in water. If any little sparkling or burning, like match-paper, be observed during this heating to redness, it may be considered as an indication of the presence of *nitric acid*—in the form of nitrate of potash, soda, or lime. In this case the loss by burning will slightly exceed the true amount of organic matter present, owing to the decomposition and escape of the nitric acid also.

* It may, for example, consist in part of geic or apocrenic acids (see *Lectures* or *Elements*) especially if there be iron in the ash which remains. Where gypsum is present in the insoluble portion, which is not unfrequently the case, the loss will be partly water—since gypsum, after being dried at 250°, loses still about 21 per cent of water when heated to redness.

The mode of estimating the quantity of this acid, when it is present in any sensible proportion, will be hereafter described.

c. The caustic potash employed to dissolve the insoluble humus (17°) takes up also any alumina which may have been in combination with the humic or ulmic acids, or may stilll remain united to the geic, crenic, mudesous, or other organic acids. When the solution is filtered, and the humic and ulmic acids are separated from it by the addition of muriatic acid till the liquid has a distinctly sour taste, this alumina, and the acids with which it is in combination, still remain in solution. After the brown flocks of humic acid, however, are collected on the filter, the alumina may be thrown down from the filtered solution by adding caustic ammonia to the sour liquid, until it has a distinctly ammoniacal smell. The light precipitate which falls must be collected on a filter, and washed with hot water till the potash is as completely separated from it as possible. It is then to be dried at 300° F., heated for some time in a close crucible over the lamp, at a temperature which *begins* to discolour it, and weighed. Being now burned in the air till it is quite white, and again weighed, the last loss may be considered as mudesous, or some similar organic acid.

The reason why this second method of drying over the lamp is here recommended, is that alumina and nearly all its compounds part with their water with great difficulty; and even with the precautions above prescribed, it is very likely that a larger per-centage of organic matter may be indicated by this experiment than in reality exists in the alumina collected and dried. The check which

the accurate experimenter has upon all these determinations is this, that the sum of the several weights of the humic and ulmic acids, the insoluble humus, the vegetable fibre—and of the crenic and mudesous acids, if present—should be somewhat less than that of the whole combustible organic mater, as determined by burning the dry soil in the open air (15°). This quantity we have seen to be in most cases greater than the truth, because any remaining water, or any nitric acid the soil may contain, is at the same time driven off.

I may further remark upon this subject, that the quantity of alumina thus dissolved by the caustic potash, is in most soils very small, and the quantity of organic matter by which it is accompanied in many cases so minute, that the determination of it may be considered as a matter of curiosity, rather than one of practical importance.

CHAPTER III.

SALINE MATTER OF THE SOIL.

Qualitative determination of the matters soluble in water.—To test for sulphuric acid, for chlorine, for alumina, magnesia, and the oxides of iron and manganese, for lime, for potash and soda, for ammonia and for phosphoric acid, by different methods.

19°. With a view to determine the *nature* of the soluble saline matter in the soil, a preliminary experiment must be made. An unweighed portion must be introduced into five or six ounces of boiling distilled water* in a flask and kept at a boiling temperature, with occasional shaking for a quarter of an hour. It may then be allowed to subside, after which the liquid is to be filtered till it passes through clear. This is to be repeated once or twice, and the several liquids mixed. The solution is then to be tested in the following manner:

Small separate portions are to be put into so many clean wine-glasses or test-tubes, and the effect produced upon these by different chemical substances carefully noted. If with a few drops of—

20°. *Nitrate of baryta*, it gives a white powdery precipitate, which does not disappear on the addition of nitric

* In all analytical operations in which the use of water is spoken of, it must be understood that pure distilled water is meant.

or muriatic acid, *the solution contains sulphuric acid.* If the precipitate does disappear on adding the acid, it contains *carbonic acid.* In this latter case, the liquid—if concentrated to a small bulk—will also effervesce on the addition of either of the acids above mentioned.*

21°. If with *nitrate of silver* it gives a white curdy precipitate, insoluble in pure nitric acid, and speedily becoming purple in the sun, it may be presumed to contain chlorine.

22°. If with *caustic ammonia* it gives a pure white *gelatinous* precipitate, it contains either *alumina,* or *magnesia,* or both. In this case, muriatic acid must be added till the precipitate disappears, and the solution is distinctly acid. If on the addition of ammonia again, and in excess, the precipitate reappears undiminished in quantity, it contains *alumina only.* If it be distinctly *less* in quantity, we may infer the presence of both magnesia and alumina; and if no precipitate now appears, that it contains *magnesia only.* If a large quantity of magnesia be present, it may be necessary to re-dissolve the precipitate thrown down by ammonia, and to make the solution acid a second time before—on the *re*-addition of ammonia—the precipitate will entirely disappear.

23°. If the precipitate, by ammonia, have more or less of a brown colour, the presence of *iron,* and perhaps *manganese,* may be inferred. If, after re-dissolving and adding ammonia a second time, the colour of the precipitate has

* The learned reader will understand why, for the sake of simplicity, I take no notice of substances not likely to be present in the soil—as, for example, baryta, which would here be thrown down along with the lime, or of oxalic acid, which, equally with the sulphuric or carbonic acid, would give a white precipitate with nitrate of baryta.

disappeared, it has been due to manganese only; if it still continue brown, it is owing chiefly or altogether to the presence of oxide of iron. If the colour of the precipitate by ammonia, be very dark, it consists almost entirely of oxide of iron, and may contain little or no alumina ; when it is only more or less brown, the presence of both alumina and oxide of iron may with certainty be inferred.

24°. *a.* If, after the first addition of ammonia, the solution be filtered to separate the alumina, the oxides of iron and manganese, and the magnesia that may be thrown down—if then, with *oxalate of ammonia*, it gives either immediately, or after a time, a white cloud, which gradually falls to the bottom in the form of a white powder, it contains *lime*. The greater and the more speedy the milkiness, the larger the quantity of lime may be presumed to be. Of course, if ammonia produces no change in the liquid, the oxalate of ammonia may be added at once, without the previous filtration, which is required to separate oxide of iron, alumina, or magnesia.

b. If oxalate of ammonia be thus added till all the lime falls, and the liquid be again filtered, evaporated to dryness, and then heated to incipient redness in the air, till the excess of oxalate of ammonia is destroyed and driven off—and if a soluble residue then remain,* it is probable that *potash* or *soda*, or both, are present.

c. If, on dissolving this residue in a little water, the addition of a few drops of a solution of tartaric acid to it produce a deposit of small colourless crystals (of cream of tartar), or if a drop of a solution of bi-chloride

* Not precipitated from its solution by ammonia, for if so precipitated, it is partly at least chloride of magnesium.

of platinum produce in a short time a yellow powdery precipitate, it contains *potash*. If no precipitate is produced by either of these—re-agents, as they are called—the presence of soda may be inferred. If a yellow precipitate, containing potash and platinum, be formed, it is to be separated by a filter, and the filtered solution, after being treated with sulphuretted hydrogen and again filtered, to separate the excess of bi-chloride of platinum, is to be evaporated to dryness. If, then, a soluble saline residue still remain, the solution contains *soda* as well as potash.

It is to be observed, that some magnesia, if present, may accompany the potash and soda through these several processes. After the separation of the potash, a little caustic ammonia will detect the presence of magnesia; but it will rarely be found so far to interfere with this *preliminary* examination as to prevent the experimenter from arriving at correct results.—(See 37°),

25°. If the addition of bi-chloride of platinum to the solution—filtered from the soil and evaporated to a small bulk—give a yellow precipitate, it contains either *potash* or *ammonia*. If, when collected on the filter, dried, and heated to bright redness in the air, white fumes are given off by this yellow precipitate, and only a spongy mass of metallic platinum remains behind, the solution contains ammonia only. If, along with the platinum, there remains after the heating, a portion of a substance which is soluble in water, has a taste like that of common salt, and gives again a yellow precipitate with bi-chloride of platinum, it contains *potash*,—and if the spongy platinum contained in the burned mass, after prolonged heating, amount to more

than 57 per cent of the weight of the yellow precipitate collected, or if it be to the matter soluble in water in a higher proportion than that of 4 to 3, the solution contains both *potash* and *ammonia*.

26°. The presence of *ammonia* among the substances extracted by water from the soil may also be detected by evaporating the liquid to a small bulk, or to perfect dryness on the water-bath, and then adding a few drops of a solution of caustic potash. The smell of ammonia, if present, becomes immediately perceptible; or if in too small quantity to be detected by the smell, it will, if present, restore the blue colour to reddened litmus paper. This experiment is best made in a small tube.

27°. If, when the solution, obtained directly from the soil, is evaporated to dryness, and the residue heated to redness in the air, a deflagration or burning like match-paper be observed, nitric acid is present. Or, if the dry mass, when put into a test-tube with a little muriatic acid, evolves distinct red fumes on being heated, or enables the muriatic acid to dissolve gold-dust, and form a yellow solution; or, if to a colourless solution of green vitriol (sulphate of iron), introduced into the tube along with the muriatic acid, it imparts more or less of a brown colour—in any of these cases the presence of nitric acid may with certainty be inferred. It will be only on rare occasions, however, that salts, so soluble as the nitrates, will be found in sensible quantity in the small portion of a soil likely to be employed in these preliminary experiments. —(See 69°.)

28° *a*. If ammonia throw down nothing from the solution, and if nothing fall when a few drops of a solution

of chloride of calcium is added, no phosphoric acid is present.

b. Or, if nothing be thrown down by ammonia, the solution may be evaporated to a very small bulk, rendered acid by the addition of nitric acid, and then dropped into a solution of molybdate of ammonia, prepared as described in 40° *a*. If any trace of a yellow precipitate now fall, phosphoric acid is present.

c. But if ammonia cause a precipitate (see 22°), and after this is separated by the filter nothing farther falls on adding the chloride of calcium as above described (*a*), the phosphoric acid, if any is present, is contained in the precipitate thrown down by ammonia. Let this, after being well washed on a filter with distilled water, be dissolved off with a little diluted nitric acid. This solution, reduced if necessary to a small bulk, is to be dropped into a solution of molybdate of ammonia, as above described (*b*), and thus tested for phosphoric acid.

d. Or, if the precipitate thrown down by ammonia be wholly or in part insoluble in pure acetic acid in the cold, that which is undissolved probably contains phosphoric acid in combination with peroxide of iron, or alumina, or both, and may be farther tested, if thought necessary, with molybdate of ammonia. But if cold acetic acid dissolve the whole, it may be inferred that no phosphoric acid is present in the precipitate thrown down by ammonia.*

I have been thus particular in describing the methods

* This latter sentence is true only in the case of a precipitate obtained by means of ammonia from a solution which, like that of a soil, always contains iron and alumina. If a solution of phosphate of lime (bone earth), or of phosphate of magnesia in muriatic acid, for example, be treated with ammonia in excess, a white precipitate will fall which does contain phosphoric acid, and yet

of detecting phosphoric acid in the soil, both because it has always been one of the most difficult substances to detect when present only in minute quantity, and because it is also one of the most important ingredients of a soil.

29°. If, when the solution is evaporated to dryness on the water-bath, the dry residue moistened with muriatic acid and treated with water—if then a light flocky matter be separated, which, when collected and heated to redness, only burns white and does not disappear, it contains *silica*.

These preliminary trials being made, notes should be kept of all the appearances presented. The arranging of the method and order to be adopted for separating from one another, and for estimating the weight of each of the substances, will depend upon the number and nature of those which the solution has been found actually to contain.

It is to be observed that the methods above described, as suited for the examination of the saline solutions obtained from a soil, are equally applicable to the examination of any other saline solution, whether natural or artificial.

dissolves readily in acetic acid. But if, before adding ammonia to the muriatic acid solution of the phosphate, a few drops of a solution of sulphate of peroxide of iron be poured into it, then ammonia throws down a precipitate of a brownish colour, which is not *wholly* dissolved by acetic acid. The peroxide of iron which was added has united with phosphoric acid, and formed a combination which is insoluble in acetic acid.

CHAPTER IV.

ESTIMATION OF THE SALINE MATTERS.

Quantitative determination of the several substances dissolved out by water.—Estimation of the sulphuric acid, of the chlorine, of the lime, of the oxide of iron, of the alumina, and of the manganese.—Different methods of estimating the magnesia.—Estimation of the potash, soda, and ammonia.—Difficulties attending the estimation of the phosphoric acid.—Method by molybdate of ammonia.—Examination of natural waters.

30°. The quantity of soluble saline matter extracted from a moderate quantity of any of our soils is rarely so great as to admit of a rigorous analysis being made, and the preceding determination of the *kind* of substances it contains will be in most cases sufficient. Cases may occur, however, in which much saline matter may be obtained by treating the soil with water.—(See 69°.) It will be proper, therefore, briefly to state the methods by which the respective quantities of each constituent may be accurately determined.

31°. *Estimation of the sulphuric acid.*—The solution being gently warmed, a few drops of nitric acid are to be added until it becomes slightly sour, and any carbonic acid that may be present is expelled. Nitrate of baryta is then to be added to the solution as long as anything falls. The white precipitate, which is sulphate of

baryta, is then to be collected on a weighed filter, well washed with distilled water, dried over boiling water, or at 212° Fahr., as long as it loses weight, and then weighed. The weight of the filter being deducted,* every 100 grains of the dry powder are equal to 34.31 grains of sulphuric acid.

32°. *Estimation of the chlorine.*—The solution of nitrate of silver must be added as long as any precipitate falls, the precipitate then washed, dried at 212° Fahr., and weighed as before. Every 100 grains of chloride of silver indicate 24.72 grains of chlorine, or 40.56 grains of common salt.

33°. *Estimation of the lime.*—A little diluted muriatic acid being now added to throw down the excess of silver, and a little sulphuric acid to separate the excess of baryta added in the former operations, and the precipitates caused by these acids separated by filtration, caustic ammonia is to be poured in, till the solution is distinctly alkaline. If no precipitate fall, oxalate of ammonia is to be added as long as any white powder appears to be produced. The solution must then be left to stand over-night, that the whole of the lime may separate; the white powder afterwards collected on a filter, washed, dried, and burned with the filter at a low red heat. The grey powder obtained is carbonate of lime, every 100 grains of which—the ash of the filter being deducted—contain 44 grains of lime.

To be quite sure that the whole of the lime is thrown

* Or the whole may be heated to redness in the air, and the filter burned away. In this case, the weight of ash left by the paper must be ascertained by previous trials, and the due proportion deducted from the weight of the sulphate (p. 18, note.)

down, it is proper to add a few additional drops of oxalate of ammonia to the clear solution before filtering it. If no milkiness is produced after half an hour or an hour, the lime has all fallen.

34°. *Estimation of the peroxide of iron.*—But if a precipitate fall on the addition of ammonia, as above prescribed, the solution may contain magnesia, alumina, and the oxides of iron and manganese. In this case the precipitate is to be re-dissolved by the addition of muriatic acid to the solution till it is distinctly acid, and ammonia again added in slight excess. If any precipitate now fall, it will consist only of alumina and oxide of iron, unless magnesia and oxide of manganese be present in large proportion, when a minute quantity of each may fall at the same time.

The precipitate is to be collected on the filter as quickly as possible—the funnel being at the same time covered with a plate of glass, to prevent the access of the air—washed with hot distilled water, and then re-dissolved in muriatic acid. This is best effected by spreading out the filter in a small porcelain dish, adding dilute acid till all is dissolved, and then washing the paper well with distilled water. A few drops of nitric acid are then to be added, and the solution heated, to convert the whole of the iron into peroxide. A solution of caustic potash added *in excess* will at first throw down both the oxide of iron and the alumina, but will afterwards re-dissolve the alumina, and leave only the oxide of iron. A gentle heat will assist this process. The oxide of iron is to be collected on a filter, washed, dried, heated to redness, and weighed. Every 100 grains of this peroxide are equal to 90 grains of

protoxide, in which state the iron had most probably existed in the original watery solution from the soil.

35°. *Estimation of the alumina.*—To the potash solution muriatic acid is added till the alkali is saturated, or till the solution reddens *litmus paper,** when the addition of ammonia precipitates the alumina; or a solution of sal-ammoniac may be added to the potash solution till the alumina falls. As it is difficult to wash this precipitate perfectly free from potash, it is better, after collecting it on the filter, to dissolve it off again by means of dilute muriatic acid, and to re-precipitate it by caustic ammonia. When well washed, dried, and weighed, this precipitate gives the true quantity of alumina present in the solution, or in the portion of saline matter submitted to analysis.

36°. *Estimation of the oxide of manganese.*—To the ammoniacal solutions from which the oxalate of lime has been precipitated (33°), a solution of hydro-sulphuret of ammonia is to be added. The manganese will fall in the form of a flesh-red sulphuret. When this precipitate has fully subsided, it must be collected on the filter and washed with water containing a very little hydro-sulphuret of ammonia. The filter is then put into a glass or porcelain basin, the precipitate dissolved off by dilute muriatic acid, and the solution warmed and filtered, if necessary. A solution of carbonate of soda then throws down carbonate of manganese, which is collected,

* Litmus paper is paper stained by dipping it in a solution of litmus, a vegetable blue colour, prepared and sold for the purpose of detecting the presence of free acids, by which it is reddened; or of free alkalies, by which, after being reddened by an acid, the blue colour is restored.

dried, and heated strongly to redness in the air. Of the brown powder obtained, every 100 grains indicate the presence of 93.03 grains of protoxide of manganese in the salt or solution under examination.

It will only be in rare cases that alumina, or the oxides of iron or manganese, will be found in sensible quantity in the watery extract of a soil. Sulphate of iron and sulphate of alumina do sometimes occur, however, in the soil, and to these unfrequent cases the processes above described are intended to apply. Of course they instruct the reader also how to separate iron, alumina, and manganese from mineral waters or any other saline solutions.

37°. *Estimation of the magnesia.*—*a.* If no potash or soda be present in the residual solution after separating the manganese, the determination of the magnesia is easy. A few drops of muriatic acid are added, and the whole gently heated, and afterwards filtered, to separate the sulphur of the excess of hydro-sulphuret of ammonia previously added. The solution is then evaporated to dryness, and the dry mass heated slowly to dull redness, to drive off all the ammoniacal salts. A few drops of diluted sulphuric acid are added to what remains, to change the whole of the magnesia into sulphate, the mass again heated to redness, and weighed. One hundred grains of this sulphate indicate the presence of 33.33 grains of pure magnesia.

b. Or after filtration, as above, to separate the sulphur ammonia is again added in excess, and then a solution of phosphate of soda. Ammoniacal phosphate of magnesia falls which is collected on the filter, washed with water

containing a little ammonia, dried and heated to redness, and weighed. Every 100 grains contain 35.94 of magnesia.

This is the converse of the process described in 40° *c*.

c. But if potash or soda be present—the weight of which it is desirable also to determine—the mode of separating the magnesia will vary according as the solution contains or is free from sulphuric acid.

If it contain no sulphuric acid, the dry mass, after heating to dull redness, is treated with a little water which dissolves it all except a little magnesia, which need not be separated. A quantity of red oxide of mercury—previously reduced to an exceedingly fine powder, by rubbing in a mortar with water—is then to be added, well mixed—the mixture evaporated to dryness, and then heated to redness. Water now dissolves out the potash and soda only, and leaves the magnesia. This is to be collected on a filter, washed—not with too much water—heated to redness, and weighed.

d. Or another way is, to prepare carbonate of silver, by precipitating nitrate of silver with carbonate of ammonia and washing—then to boil the solution of the mixed chlorides with this carbonate of silver till the solution becomes strongly alkaline. When filtered hot, only magnesia and chloride or carbonate of silver remain on the filter. A little dilute muriatic acid poured upon the washed filter dissolves out the whole of the magnesia. The solution evaporated nearly to dryness, treated with a few drops of sulphuric acid, then again evaporated to dryness and heated to redness, gives the magnesia in the

state of sulphate, of which every 100 grains contain 33.33 grains of magnesia.

c. But if sulphuric acid be present along with the magnesia, potash, and soda, we dissolve the dry mass in water, adding a little muriatic acid, if necessary, to dissolve the magnesia, which may have become insoluble in water. Ammonia is now poured in, in excess, and, if this cause a milkiness, a little more acid, till the addition of ammonia, again in excess, leaves the solution clear. Arseniate of ammonia now throws down the magnesia, which, after twelve hours, may be collected on a filter, washed with cold water which contains and smells of ammonia, and then dried at 212° Fahr. Every 100 grains of the precipitate thus dried contain 21 grains (20.97) of magnesia. This method separates the magnesia, but not the sulphuric acid, from the solution.

f. Or, when sulphuric acid is present, baryta water may be added to the solution, which will precipitate the sulphuric acid as sulphate of baryta, and the magnesia at the same time as caustic magnesia. It is quickly filtered and washed. Dilute sulphuric acid then dissolves the magnesia from the filter, leaving the sulphate of baryta. The solution of magnesia is evaporated, and the sulphate of magnesia determined as in *a*.

38°. *Estimation of the potash and soda.—a.* If no sulphuric acid has been present, the solution containing the potash and soda, as left by process 37° *c*, is to be evaporated to dryness, and heated to dull redness. The weight of the mass, which consists of a mixture of chloride of potassium with chloride of sodium (common

salt), is accurately determined; it is then dissolved in a small quantity of water, and a solution of bi-chloride of platinum added to it in sufficient quantity. Being evaporated by a very gentle heat nearly to dryness, weak alcohol is added which dissolves the chloride of sodium and any excess of salt of platinum which may be present. The yellow powder is collected on a weighed filter, washed well with alcohol, dried by a gentle heat, and weighed on the filter. Every 100 grains indicate the presence of 19.31 potash, of potash, or 30.56 grains of chloride of potassium.

The quantity of chloride of sodium is estimated from the loss. The weight of the chloride of potassium above found is deducted from that of the mixed chlorides previously ascertained: the remainder is the weight of the chloride of sodium. Every 100 grains of chloride of sodium (common salt) are equivalent to 53.17 of soda.

b. When the magnesia has been separated by means of silver, the solution, before being evaporated, is to be made slightly sour with muriatic acid, that the chloride of silver may be thrown down, collected on a filter, and washed. The filtered solution is then evaporated and treated as in *a*.

c. When the magnesia has been separated by means of arseniate of ammonia, the solution containing the alkalies is to be evaporated to dryness, and heated to redness in a *porcelain* crucible, till fumes cease to be given off. When cold, add a little sal-ammoniac, and heat again. By this operation the excess of arsenic acid is wholly driven off, and, as the fumes of this arsenic acid are poisonous, care must be taken that they are not inhaled. The mixed chlorides of potassium and sodium remain as in the

method *a*, and are to be treated in the same way, if no sulphuric acid is present. But if sulphuric acid is present, the mixed salts are dissolved in water, and the acid precipitated by baryta water, and separated by filtration, as in 37 *f*.

d. After the precipitation by baryta, according to this method (37 *f*) ammonia and carbonate of ammonia are added to the filtered solutions which are heated to boiling. In this way the excess of baryta is separated. The filtered solution is then evaporated to drive off the ammonia, muriatic acid added to convert the alkalies into chlorides, and then these mixed chlorides separated as described under *a*.

39°. *Estimation of the ammonia.—a*. If ammonia be present in the solution along with potash and other substances, the method by which it can be most easily estimated is to introduce a separate portion of the solution into a large tubulated retort, to add water until it amounts to nearly an English pint—then to introduce a quantity of caustic potash or caustic baryta, and to distil by a gentle heat into a close receiver, containing a little dilute muriatic acid, until fully one-half has passed over. Bi-chloride of platinum is then to be added to the solution which has come over, previously rendered slightly acid by muriatic acid, and the whole is evaporated *nearly* to dryness by a very gentle heat. Dilute alcohol is then added to wash out the excess of the salt of platinum, and the yellow powder is collected on a filter, washed with spirit, dried by a *very* gentle heat and weighed. One hundred grains indicate the presence of 7.61 grains of ammonia.

b. Or the yellow powder, without being so carefully dried, may be heated to redness, when only metallic platinum will remain. One hundred grains of this metallic platinum indicate the presence of 17.21 grains of ammonia.

c. Or the acid liquor in the receiver may be evaporated to dryness in a glass beaker over the water-bath till it ceases to lose weight. The salt obtained is sal-ammoniac, of which every 100 grains represent 31.8 grains of ammonia. But this method is not so accurate as the preceding.

40°. *Estimation of the phosphoric acid.*—The presence of phosphoric acid in the solution being already ascertained (28°), its quantity may be most easily estimated as follows:—

a. Dissolve *molybdic acid* in ammonia, and to the solution add pure nitric acid till the precipitate at first formed again dissolves. This acid solution of molybdate of ammonia is the most delicate test for phosphoric acid yet known. The substance to be examined by it for phosphoric acid may be dissolved in muriatic acid, but nitric acid is to be preferred. Or, if the substance be already in solution, as in the watery extract of a soil, the solution, as I have already directed (28°), should be made acid by means of nitric acid.

b. Evaporate the solution to be tested to a small bulk, and add to it a *large excess* of the solution of molybdate of ammonia. This is necessary, because the yellow precipitate which falls contains nearly 30 times as much molybdic acid as it does of phosphoric acid. It is known that enough of the molybdic has been used when, before col-

lecting the precipitate on a filter, a drop of the supernatant liquid gives, with sulphuretted hydrogen water, a brown precipitate of sulphuret of molybdenum.

Digest the solution with the yellow precipitate which has fallen, for several hours, at a gentle heat; collect the precipitate on a filter, and wash it with a fresh portion of the same solution of the molybdate as was employed to throw it down. This precipitate on the filter contains all the phosphoric acid. But to be sure of this, the filtered solution (filtrate) should be set aside for some time in a warm place, to see if any farther precipitate falls.

c. The yellow precipitate upon the filter is now to be dissolved, while still moist, in solution of caustic ammonia, and washed well from the filter. To this new solution add one of ammoniacal sulphate of magnesia as long as a precipitate is seen to fall. Set aside for twelve hours in the cold. The ammoniacal phosphate of magnesia which has fallen is in the state of minute shining crystals, which can be well distinguished by the aid of a pocket microscope. It is to be collected on a filter, washed with water, with which a third or fourth part of solution of ammonia has been mixed, dried, heated to redness, the filter burned separately, and its ashes added to the rest. Deducting the ashes of the filter (p. 18, note), every hundred grains of the phosphate of magnesia thus obtained contain 64.06 grains of phosphoric acid (note, p. 74).

d. The ammoniacal sulphate of magnesia required in this operation is easiest prepared by dissolving sulphate of magnesia in water, adding ammonia to throw down magnesia, then muriatic acid to dissolve it; again ammonia—followed by muriatic acid, sufficient, on shaking, to make

it clear again—and so on, till the addition of ammonia *in excess* no longer makes the solution milky. The preparation may be made more quickly by adding at once a solution of sal-ammoniac* sufficient to prevent ammonia from throwing down the magnesia; but in this way the young manipulator is more likely to add an excess of sal-ammoniac, which is to be avoided.

The process above described is excellent for determining small quantities of phosphoric acid, such as occur in a soil. The quantity of molybdic acid required, in proportion to the phosphoric acid, is so great, however, that when larger quantities of phosphoric acid are to be determined, it is usual to employ other methods, which need not be here described.

Note on the examination of waters.—It may be of use to some of my younger readers, if I here remark that natural waters are examined in the same way as the watery solution of a soil. The methods described in the two preceding chapters for the qualitative and quantitative examination and analysis of an artificially prepared solution, will enable the young experimenter to arrive at satisfactory results in regard to all but a few of the more rare natural solutions which form our springs. The ochrey or calcareous deposits which sometimes fall from them, either when exposed to the air, or when they are boiled, must be treated like the earthy matter of the soil, as described in the succeeding chapter.

* The reader understands that the ammonia and muriatic acid, successively added, form sal-ammoniac in the solution itself.

CHAPTER V.

EARTHY MATTERS OF THE SOIL.

Matter insoluble in water.—Treatment with muriatic acid; testing of this solution.—Estimation of the soluble silica, the sulphuric acid, the iron as peroxide, the alumina, lime, magnesia, oxide of manganese, phosphoric acid, potash and soda.—How to estimate the protoxide of iron and the carbonic acid in a soil.—Treatment of substances insoluble in muriatic acid.—Heating with sulphuric acid—Fusion with alkaline carbonates.—Summary of the preceding methods.—Note on tile and fire clays.

§ I. ESTIMATION OF THE SUBSTANCES SOLUBLE IN MURIATIC ACID.

41°. An unweighed portion of the soil in its natural state has been used for the preparation of the watery extract already examined, as described in Chapter III. A part of this same soil, after being so extracted with water, is to be treated with muriatic acid diluted with one or two waters. If bubbles of gas are seen to escape from the soil when the acid is first added, the presence of carbonate of lime, and in some cases of carbonate of magnesia, may be inferred. The quantity of carbonate may be judged of by the rapidity and length of time during which these bubbles of gas continue to be given off.

42.° The soil and acid thus mixed are to be disgested on a sand or water bath for some hours, diluted with water and the solution filtered. It is then to be examined qualitatively, as described in Chapter III. Generally it will be found to contain silica, alumina, oxide of iron, oxide of manganese, lime, magnesia, potash and soda, with sulphuric and phosphoric acids. But some of these may be absent; and this it is necessary to ascertain, as it will then be unnecessary in the subsequent analysis to take any steps for determining their quantity. The silica may be sought for as described in 45°.

43°. *a*. This preliminary examination being made, a weighed portion of the soil, dried at 250° Fahr., and weighed, is first repeatedly boiled in distilled water, as described in 19°, and then covered over with diluted muriatic acid, and gently heated on a water or sand bath, with occasional stirring for twelve hours. Water is now added, the whole well shaken or stirred together, allowed to settle, and the solution poured off through a filter.

b. Over the still undissolved portion of the soil strong muriatic acid may now be poured so as just to cover it, and the whole disgested again, with the aid of heat, for several hours. Water is added, the whole thrown upon the filter already employed (*a*), and the insoluble matter washed till water ceases to remove anything, and dried at 250° Fahr., till it ceases to lose weight. This weight, deducted from that of the dry soil employed, indicates the proportion of the washed soil which is soluble in acids.

From 100 to 400 grains of the dry soil may be taken for this treatment with acid, according as it naturally contains more or less sand and gravel.

44°. The dry insoluble matter being now heated to redness, the organic matter burns away, and there remains only the insoluble *mineral* matter of the soil. This is to be weighed, and kept for subsequent treatment and analysis. The loss, which is organic matter insoluble in water or acid, may probably serve as a check upon some of the determinations already made, when examining the soil in reference to its organic matter only, as described in Chapter II.

45.° To the mixed acid solutions, 43° *a* and *b*, which will probably be of a reddish-brown colour, from the presence of peroxide of iron, a little nitric acid is to be added, after which they are to be evaporated to dryness on the water-bath, moistened with muriatic acid, and, after standing some time, treated with water. The silica remains insoluble, and may be collected on a filter, washed, dried, heated to redness, and weighed. The weight indicates the proportion of silica existing in the soil in a state to be readily dissolved by acids.

In less careful analysis of soils, this separation of the silica soluble in acids, which is rarely large in amount, may be neglected.

46°. The solution filtered from the silica contains all the ingredients of the soil which are soluble in muriatic acid, and the nature and names of which have been already determined (42). These ingredients may be separated from each other, and their several weights estimated most readily, as follows. If all the substances named in 42° have been found in it, divide the solution into *four* equal parts by measure.

47°. In the *first*, estimate the sulphuric acid—if any be

present—by adding to it a solution of nitrate of baryta or of chloride of barium, and treating the precipitate as described in 31°. If no sulphuric acid be present, the solution need only be divided into three parts.

48°. In the *second*, determine the phosphoric acid by means of molybdate of ammonia, as described in 40°. With this view, it may be advantageously evaporated to dryness, and treated with concentrated nitric acid aided by heat, till the smell of chlorine passes off. It may then be dissolved in a little diluted nitric acid and added to the solution of molybdic acid. Or the peroxide of iron and alumina may be at once precipitated from the solution, collected on a filter, dissolved while moist in dilute nitric acid, and added to the molybdate of ammonia. As I have already said, the whole of the phosphoric acid contained in a soil almost invariably falls along with the peroxide of iron and the alumina.

49°. From the *third* solution may be determined—

a. The *lime and magnesia*, as described in 33° and 37°.

b. The *peroxide of iron*, as described in 34°.

c. The *alumina*, as described in 35°. The alumina obtained in this way will contain also the phosphoric acid. But the weight of the latter has already been determined in the second portion of liquid (48°), and this, deducted from the weight of the alumina here found, will give the true weight of the alumina.

In some cases a portion of the phosphoric acid may remain in the oxide of iron which has been separated from the alumina, but the proportion of phosphoric acid in a soil is generally so small that, in an analysis of this

kind, it is rarely necessary to have recourse to further refined manipulations for the sake of a theoretical accuracy which can lead to no practical result.

But it may happen also that the soil will contain so much phosphoric acid, that, when ammonia is added to the acid solution, a quantity of phosphate of lime or phosphate of magnesia, or both, may fall along with the iron and alumina. This may be tested as follows :—

Dissolve the precipitate in sulphuric acid, add an equal quantity of sulphate of ammonia, making a clear solution with the smallest possible quantity of water. Into this solution pour alcohol in *large* quantity, shake well together, and leave the whole to settle. Add now a little ether, and if this troubles the solution, let it stand again for twelve hours, then filter and wash the precipitate with a mixture of alcohol and ether. Distil off the alcohol and ether, and test the remaining solution for phosphoric acid by means of ammoniacal sulphate of magnesia, as described in 40° *c*.

The precipitate on the filter will dissolve in water, from which ammonia will throw down oxide of iron and alumina---oxalate of ammonia, lime, and phosphate of soda, magnesia, if any of these are present.

If by this testing the presence of lime and magnesia in the precipitate from the acid solution of the soil by ammonia, be ascertained—the phosphoric acid may be separated from it (the precipitate), and estimated as phosphate of magnesia in the way here described---the iron and alumina as described in 34° and 35°---and the lime and magnesia as described in 33° and 37°.

d. The oxide of manganese may be determined as de-

scribed in 36°, though in ordinary soil analysis the special separation of this oxide may be neglected, as it is usually present only in small quantity

e. Potash and soda, also, will only be present in very minute quantity in this acid solution, and in most soil analysis may be neglected. In this case the magnesia is thrown down at once by phosphate of soda (37° *b*.) If it be desired accurately to estimate them, however, either together or singly, it may be done by the methods described under 38°.

50°. From the *fourth* solution, the total quantity of iron may again be determined by the *measure* method hereafter described under 65°. Or if this trial, which is very brief and simple, be thought unnecessary, the acid solution may only be divided into three portions (46°). There is an advantage, however, in such a double determination by independent methods, as the one result serves to control and test the other.

51°. *Estimation of the protoxide of iron* in the soil. It is known that there are two oxides of iron.

	Iron.	Oxygen.
The first or protoxide, consisting of	56	16
The second or peroxide, or sesquioxide	56	24

The first of these, by exposure to the air, soon changes into the second. Nevertheless, it frequently occurs in the soil, and sometimes materially affects its agricultural qualities. It is necessary, therefore, in many cases, that the quantity of this protoxide contained in a soil should be accurately estimated. By the process described in 34°, or by the measure method 65°, the *whole* of the iron has been already obtained in the state of peroxide. The fol-

lowing method enables us to determine how much of this existed in the soil in the state of *prot*oxide :—

A little powdered chalk or common soda is introduced into a flask with a longish neck, diluted muriatic acid poured upon it, and allowed to stand till it has *all* dissolved, and the flask has become filled with carbonic acid gas. A weighed portion of the dry soil is then to be introduced into the flask, diluted muriatic acid poured over it—not in too large excess—the flask lightly corked, and the whole digested till the oxides of iron are all dissolved. Powdered carbonate of baryta is now to be introduced *in excess*, and the whole digested with the aid of occasional shaking. By the action of the baryta, the whole of the peroxide of iron and alumina will be gradually separated from the solution, while the protoxide of iron will remain dissolved. The full production of this effect will be indicated by the solution becoming nearly colourless, or only of a pale green. The flask is now to be filled with hot *boiled* distilled water, corked carefully, and the whole allowed to settle. The solution is then decanted, or, if necessary, filtered, and the insoluble matters collected on the filter and washed. The solution contains all the iron which existed in the soil in the state of protoxide. It is boiled with a little nitric acid to convert it into peroxide, precipitated by ammonia, and separated from alumina, if any be still present, by the method already described in 34°. When dried and weighed, every 100 grains of this peroxide represent 90 grains of protoxide in the soil.

The weight of the peroxide thus obtained, deducted from that of the whole peroxide of iron already determined, 49°*b*, gives the weight of peroxide actually existing in the soil.

In this process the flask is filled with carbonic acid, only to prevent the protoxide from absorbing oxygen. Where carbonate of baryta cannot be obtained, carbonate of lime or chalk may be used in its stead.

52°. *Estimation of the carbonic acid in the soil.*—When lime and magnesia are present in a soil, they are usually combined for the most part with carbonic acid. A smaller portion is in combination with humic and hulmic acids, and a portion probably also with silica. When the soil is treated with muriatic acid, these carbonates of lime and magnesia are decomposed, and their acid escapes in the form of carbonic acid gas.

It is not usual, in the analysis of a soil, to estimate directly the weight of this carbonic acid. The lime and magnesia obtained in an analysis are commonly considered to have been altogether in the state of carbonates in the soil—except what is known to have been in the states of sulphate or phosphate—and the carbonic acid required to convert them into carbonates is calculated and adopted as the true proportion of this acid contained in the soil. This is not strictly correct, however; and where rigid accuracy is required, it is necessary to estimate the weight of the carbonic acid directly. This may be done by the following method:—

a. One or two hundred grains of the soil, carefully dried at 212° to 250° Fahr., are to be introduced into a small weighed flask or bottle, and then just covered with a weighed quantity of cold diluted muriatic acid. The carbonates in the soil will be gradually dissolved, and the carbonic acid will be set free in the form of gas. After twelve hours, or when the action has entirely ceased, a

small tube is to be introduced through the mouth of the flask nearly to the surface of the solution, and air sucked through it till the whole of the carbonic acid is drawn out of the flask. The flask with its contents is now weighed, and the loss of weight indicates the amount of carbonic acid very nearly. Every 100 grains of carbonate of lime in the soil will lose in this way 44 grains of carbonic acid—or every 100 grains of carbonic acid indicate the presence of 227.27 grains of carbonate of lime, or of 127.27 grains of lime in the state of carbonate.

b. This method is not rigorously accurate, since, on the one hand, a small loss of weight may occur from the escape of watery vapour carried off by the gas or sucked out by the tube; and, on the other, a small gain, by the retention of a small quantity of carbonic acid in the solution itself. It is made rigorous by passing the suction or escape tube through a well-fitting cork, putting it into the flask before the commencement of the operation, and weighing them together. The cork is removed to introduce the materials, and immediately replaced. A weighed tube, filled with chloride of calcicum,* is then attached, by an India-rubber joining,† to the escape tube, so that the gas passes through it and escapes dry. By heating the solution

* Chloride of calcicum is prepared by dissolving lime in muriatic acid, evaporating to dryness, heating in a crucible till it melts, pouring it out upon a cold flag or iron plate, breaking into pieces, and, while still hot, putting it into a well-stoppered bottle. It absorbs moisture with great rapidity, so that if moist air be made to enter one end of a tube 6 or 8 inches long filled with broken pieces of the chloride, it will escape at the other end quite dry.

† Caoutchouc tubes of various sizes are prepared and sold for the purpose of connecting pieces of apparatus together. They are recommended by their softness (when gently warmed), by their elasticity, and by their power of resisting the action of acids and alkalies.

at the close of the operation, any gas retained by the liquid is also expelled; and, by slightly loosening the cork, air may be sucked through the united tubes till all the gas is drawn out of the flask. The chloride-of-calcicum tube is now detached and weighed separately. The increase of its weight, if any, is to be added to that of the flask and suction-tube weighed together.*

§ II.—ESTIMATION OF THE SUBSTANCES INSOLUBLE IN MURIATIC ACID.

53°. The part of the soil which remains insoluble in muriatic acid consists chiefly of quartz sand, of fragments of rock, and of undecomposed clay and other silictates.

If there be much pure sand and fragments of undecomposed rock, these had better be washed or picked out and weighed separately. The fine part—or the whole if thought advisable—after being reduced to fine powder, first in a steel and then in an agate mortar, may then be treated in one or other of two ways.

54°. *First.* It may be drenched with concentrated sulphuric acid, and heated for a considerable time in a slightly-covered platinum crucible over a lamp, till the sulphuric acid is nearly all driven off. When cold, it is treated with water, which, after being allowed to settle, is filtered. If, on the addition of ammonia in excess to this solution, a precipitate of alumina or of oxide of iron

* Various modifications of this apparatus are in use, some more and some less complicated. The passage of another tube through the cork, nearly to the bottom of the flask—by which the acid may be poured in at the beginning, and the air enter at the close of the operation—is an improvement, but what I have described in the text will, in careful hands, give very good results.

appears, the treatment with sulphuric acid is to be repeated a second, and if necessary, a third time, till everything soluble is taken up by the sulphuric acid.

By this treatment the insoluble silictates are decomposed, and only pure silica remains in the crucible undissolved. This may be collected on the filter, washed, dried, heated to redness, and weighed. Its weight represents the proportion of insoluble silicious matter in the soil.

The solution in sulphuric acid may contain alumina, oxide of iron, lime, and magnesia. These are to be separated, and their several weights determined by the processes already described. Only in rare cases will this solution contain potash or soda, or phosphoric acid; but if the presence of these substances be suspected, they also can be sought for and separated as described in the preceding section.

55°. *Second.—a.* Or the portion of soil on which hot muriatic acid refuses to act may be mixed with three times its weight of a mixture of carbonate of potash with carbonate of soda, in equal equivalents,* and heated to fusion in a platinum crucible. Place the crucible while still hot on a cold plate, the fused mass will then readily separate from the sides of the crucible as it cools, and can be easily taken out. The fused mass, along with the crucible, is then put into a beaker glass, covered with water

* That is, in the proportion of 69 of *dry* carbonate of potash to 53 of *dry* carbonate of soda, since these two salts consist respectively of—

Carbonate of Potash.		Carbonate of Soda.	
Carbonic acid,	22.0	Carbonic acid,	22.0
Potash, . .	47.2	Soda, . . .	31.2
	69.2		53.2

—See note, p. 82.

and slowly treated with diluted muriatic acid, aided by a gentle heat, as long as anything is dissolved. A tolerably large vessel should be employed, that nothing may escape during the effervescence that takes place. The residue is silica, which is to be collected on a filter, washed, dried, and weighed.

b. The solution in muriatic acid may contain silica, alumina, oxide of iron, lime, and magnesia. A little nitric acid being added, it is to be evaporated to dryness on the water-bath. As it approaches to dryness, it should be constantly stirred, that it may be obtained in the form of a dry powder. It is then moistened with muriatic acid, after a little treated with water, and the silica which remains undissolved collected on a filter, washed, heated to redness, and weighed. This weight is to be added to that of the silica already obtained (*a.*)

From the acid solution the alumina, oxide of iron, lime, and magnesia—or such of them as are present—are to be separated by the processes described in the preceding section.

56°.—*a. Potash and soda* will rarely be extracted from the insoluble part of the soil by this process. But if it is desired to seek for, or to estimate the amount of, these substances in the portion of the soil left undissolved by muriatic acid, it must be reduced to very fine powder and intimately mixed with four or five times its weight of hydrate of baryta, and heated to incipient fusion over a lamp in a *silver* crucible. Platinum is slightly attacked by caustic baryta. When cold, the fused mass is treated with diluted muriatic acid, as in 55° *a.*

b. The silica, soluble and insoluble, is then separated as after the fusion with potash and soda (55° *b*), and the

alumina and oxide of iron thrown down by ammonia. Sulphuric acid is then added, so as *exactly* to precipitate the whole of the baryta held in solution, after which the lime is thrown down as usual by ammonia and oxalate of ammonia. Magnesia, potash, and soda, if all present, are now in the solution in the state of chlorides, and may be separated by the methods already described.

c. But if, in separating the byrata, an excess of sulphuric acid has been added, the solution containing the magnesia and the alkalies is to be evaporated to dryness, and heated to redness, to drive off the ammoniacal salts. Dissolved in water, with the aid of a few drops of muriatic acid, if necessary, a solution of caustic baryta is to be added, by which the sulphuric acid and the magnesia are both thrown down, and may be collected on a filter and washed. Sulphuric acid dissolves out the magnesia, which may be evaporated and estimated in the state of sulphate of magnesia, as already described under 37° *f*.

d. From the solution containing the alkalies the excess of baryta is to be thrown down as carbonate, by adding carbonate of ammonia, and boiling (38° *d*.) Filtered from this, evaporated to dryness, and heated—a few drops of muriatic acid being added, if necessary, to insure their being in the state of chlorides—the alkalies are obtained alone, and may be weighed. The potash is then estimated by means of bi-chloride of platinum, as already described under 38°, and the soda from the loss.

This process I have thought it necessary to describe ; but, as I have already stated, it will very rarely be necessary to test for, much less to estimate quantitatively, the

alkaline matters contained in the portion of a soil upon which muriatic acid ceases to act.

The accuracy and care with which the successive processes have been conducted, is tested by adding together the weights of the several substances that have been separately obtained. If this sum does not differ more than one per cent from the weight of the soil employed, the results may be considered to be deserving of confidence. One of the points in which a beginner is most liable to err is in the washing of the several precipitates he collects upon his filters. As this is a tedious operation, he is very likely to wash them at first only imperfectly, and thus to have an excess of weight when his quantities are added together—whereas a small loss, in a correct analysis, is almost unavoidable. The precipitates should always be washed with distilled water, and till a drop of what passes through leaves no stain, when dried, upon a bit of glass or of bright platinum foil.

§ III. SUMMARY OF THE PRECEDING METHODS.

57°. The following scheme may be useful as giving a brief view of the successive steps which are to be taken in order to separate the several substances from the solutions in muriatic acid by the methods above described.

A°. Digest the soil in distilled water, dry at 250° Fahr., weigh, digest with dilute muriatic acid for twelve hours, and then with concentrated muriatic acid. Dilute, filter, and mix the solutions.

B°. Add a little nitric acid, evaporate to dryness,

moisten with muriatic acid, treat with water, filter, and then collect, wash, heat to redness, and weigh the silica.

C°. Divide the solution into three equal portions.

a. In the *first*, estimate the sulphuric acid by means of chloride of barium (31°).

b. In the *second*, estimate the phosphoric acid by molybdate of ammonia. (40° *b*).

c. Treat the *third* as follows: It may contain alumina, oxide of iron, oxide of manganese, lime, magnesia, potash, and soda, as well as the sulphuric and phosphoric acids already estimated in the other portions of the solution.

a. Add caustic ammonia. The precipitate contains alumina, oxide of iron, phosphoric acid, and perhaps some phosphate of lime and phosphate of magnesia. Collect on the filter, and wash.

b. Dissolve the precipitate in a small quantity of sulphuric acid, add a little sulphate of ammonia, and then pour the solution into a large quantity of alcohol. Add a little ether, filter, and wash the precipitate with mixed alcohol and ether.

c. From the solution distil off the alcohol, dilute the water, and precipitate the phosphoric acid by ammoniacal sulphate of magnesia (40° *c*). Compare the weight of this phosphoric acid with that obtained already by molybdate of ammonia C° *b*). It ought to be a little less, as by the present process the whole of the phosphoric acid is not rigorously separated.

d. Dissolve in water what remains on the filter (*b*); add caustic potash in excess, and heat. The alumina and oxide of iron with lime and magnesia, if present, are precipitated, but the alumina is re-dissolved. Filter, wash the precipitate, dry, heat to redness, and weigh.

e. Dissolve this precipitate in muriatic acid, throw down the oxide of iron by ammonia, filter, wash, dry, heat to redness, and weigh the oxide of iron again. If it weighs less than the whole precipitate did, the filtered solution contains lime or magnesia. Add it to the solution from *a*.

f. To the potash solution *d* add sal-ammoniac, and boil to precipitate the alumina. Collect on a filter, wash, heat to redness, and weigh.

Note.—If no phosphoric acid be present, or if much oxide of iron be present along with it, the pre-

cipitate from *a* by ammonia contains no lime or magnesia, and the steps *b*, *c*, and *e* become unnecessary.

g. To the ammoniacal solution from *a* (mixed, if necessary, with that from *c*), add oxalate of ammonia. Collect, wash, heat to redness, and weigh the carbonate of lime (33).

h. Add hydrosulphuret of ammonia to throw down the manganese as sulphuret. Collect, dissolve in muriatic acid, precipitate by carbonate of soda, collect again, wash, dry, heat to redness, and weigh the oxide of manganese (36°).

i. If no potash and soda are present, precipitate the magnesia by phosphate of soda (from the filtered solution *h*), collect, wash, dry, and weigh (37° *b*). Or evaporate to dryness, add a few drops of sulphuric acid, heat to redness, and weigh the sulphate of magnesia (37° *a*).

k. Or if the alkalies are present, and no sulphuric acid, evaporate to dryness, and drive off all the ammoniacal salts by heat. Dissolve them in a little water, add finely-divided red oxide of mercury, evaporate to dryness, and heat to redness. Water then dissolves the alkalies, and leaves the magnesia, which is collected and weighed (37° *c*).

l. The solution contains the chlorides of potassium and sodium. Evaporate to dryness, heat to dull redness, and weigh. Redissolve in water, separate the chloride of potassium by bi-chloride of platinum, collect, dry, and weigh (38° *a*). The chloride of sodium is estimated by the loss.

m. But if sulphuric acid be present, precipitate by caustic baryta, filter, and wash. Dissolve the precipitated magnesia from the filter by means of sulphuric acid, evaporate to dryness, and estimate either as sulphate or as phosphate. (37° *f*).

n. To the solution filtered from the baryta and magnesia, add carbonate of ammonia, and boil. Collect the carbonate of baryta on the filter, evaporate the solution to dryness, heat to drive off the ammoniacal salts, add a few drops of muriatic acid, and heat again (38° *d*).

Weigh now the mixed chlorides, and proceed as under *l*.

o. Finally, from the solution of a separate portion of the soil in muriatic acid, precipitate the peroxide of iron by means of carbonate of baryta, and then estimate the protoxide as described under 51°.

Note on tile and fire clays.—I simply add here, in reference to clays of every kind, that they are examined and analysed exactly in the same way as the insoluble matter of a soil.

CHAPTER VI.

ANALYSIS BY MEASURE—ORES OF IRON.

Estimation of substances by measure; principle on which the method is based.—Estimation of chlorine in this way.—Estimation of silver.—Estimation of the oxides of iron by measure.—Standard solution of per-manganate of potash; its effect on the protoxide of iron; mode of using it.—Estimation of the protoxide of iron in a soil.—Estimation of the whole quantity of iron.—Analysis of the ores of iron.—Estimation of the iron only, very brief and easy.—Estimation of the other ingredients longer and more difficult.

§ I. ESTIMATION OF SUBSTANCES BY MEASURE.

58°. Besides the methods already described, by which the ingredients of a soil are severally separated, collected, and directly weighed, another general method of analysis exists by which the separation and weighing of each is dispensed with, and their several quantities estimated by measure. This method, being more speedy, involving less labour, and rendering the use of the balance less frequent, has recently come much into favour, and is daily becoming more improved and more widely applicable.

This method is simply an extension of the processes of testing or qualitative examination already described in Chapter III., and is based upon the two principles—

1°. That the presence of one known body, A, may be detected in a solution by the visible change or reaction

produced in the liquid by the addition of a solution of another known body, B.

2°. That the quantity or weight of the body A may be calculated from the *known* quantity of the body B, which it is necessary to add to the solution before the visible change or re-action ceases to be produced by new additions.

59°. And the way in which these principles are applied to quantitative analysis is to prepare a *standard solution* of the body B, every cubic inch or other measure of which contains a known weight of B. If of this solution it be found necessary to add a known measure to the solution A before visible change ceases, then the weight of B being known from the measure, the weight of A, which is equivalent to it, can be calculated.

60°. Thus a solution of common salt forms a white visible curdy precipitate when added to a solution of nitrate of silver. The curdy precipitate is chloride of silver. If the solution of common salt be added drop by drop, shaking, and allowing the whole to settle after each addition, we shall arrive at length at a point when a further drop of the salt solution will produce no further precipitate.

Now, 58.46 grains of common salt (chloride of sodium), containing 35.46 of chlorine, throw down exactly 143.46 grains of chloride of silver, containing also 35.46 grains of chlorine united to 108 grains of silver.* Or 100 grains

* These two substances consist respectively of—

Common Salt.		Per cent.	Chloride of silver.		Per cent.
Chlorine,	35.46	60.663	Chlorine,	35.46	24.724
Sodium,	23.00	39.337	Silver,	108.00	75.276
	58.46	100.00		143.46	100.00

of common salt throw down 245.34 grains of chloride of silver, containing 184.68 of silver and 60.66 of chlorine. If, therefore, we dissolve 100 grains of pure common salt in distilled water, and then add water till the solution fills exactly 100 measures in our graduated tube, every measure will represent 1 grain of common salt. Suppose now we add this solution (A) to that which contains silver (B), till it ceases to throw down anything, and that on looking at our graduated tube we find that 10 measures of the common salt (A) have been required. These ten measures contain 10 grains of common salt, and represent 14.346 grains of chloride of silver, or 10.797 grains of silver, which, without weighing, is the quantity of silver contained in the solution B.

61°. Or the process may be reversed. The standard solution (A) may in every hundred measures contain 100 grains of pure nitrate of silver, and this may be employed exactly in the same say to estimate the quantity of chlorine in a solution B. Every 100 grains of nitrate of silver throw down 84.38 grains of chloride of silver containing 25.8 grains of chlorine ;* so that if exactly 10 measures of the silver solution A. be added to the chlorine or common salt solution B, before precipitation ceases, then the quantity of chlorine contained in the solution was exactly one-tenth of 25.8, or 2.58 grains.

This method may be employed, if thought desirable, in

* Nitrate of silver consists of—

						Per cent.
Nitric acid.	54.	or	1 Nitrogen,	14.	or	8.23
Oxide of silver,	116.		6 Oxygen,	48.		28.24
			1 Silver,	108.		63.53
	170.			170.		100.

estimating the quantity of chlorine in the watery solution of a soil, or in any other solution, instead of the more tedious one of collecting and weighing as described in 32°.

§ II. ESTIMATION OF THE OXIDES OF IRON BY MEASURE.

62°. The oxides of iron, and especially the protoxide of iron, may be very conveniently estimated by measure.

a. The first step is to prepare a standard solution of per-manganate of potash. For this purpose a portion of the per-manganate is dissolved in water in a stoppered bottle, is allowed to settle till quite clear, and is then decanted carefully, or is at once filtered through asbestos into another bottle which is kept well closed. The solution is of a beautiful violet colour. This solution has the property of imparting oxygen to the protoxide of iron, converting it into the peroxide, and at the same time becoming itself colourless. By adding a solution of known strength, therefore, to one containing protoxide of iron—very carefully, and with constant stirring—as long as the colour disappears, the quantity of the protoxide can be calculated.

63°. The next step, therefore, is to ascertain the strength of the solution of per-manganate of potash. For this purpose 10 grains of fine pianoforte wire are dissolved, by the aid of heat, in pure muriatic acid slightly diluted, then boiled, and distilled water added to raise the bulk of the solution to 100 measures of our graduated vessel. Every 10 measures now contain 1 grain of iron—equal to 1.285 grains of protoxide, or 1.428 of peroxide of iron.

To 10 measures of this solution, containing one grain

of iron (or a larger quantity may be taken), add the solution of the per-manganate till the mixture begins to exhibit a pale red tint, which does not disappear on stirring, and mark exactly how many measures of the per-manganate have been employed. Suppose 20 measures have been required, then we conclude that 20 measures of the solution are capable of per-oxiding one grain of iron in the state of protoxide, and therefore indicate the presence of 1.285 grains of protoxide.

This testing should be repeated two or three times, that the strength of the solution may be accurately ascertained; and it should again be repeated at intervals when, after a time, it is again to be employed, as the dissolved per-manganate gradually decomposes, and the solution consequently becomes weaker.

64°. *To estimate the quantity of protoxide in a soil*, by this method, is now easy. To the solution of a weighed quantity of the soil, prepared in an atmosphere of carbonic acid, as described in 51°, and diluted with water, add that of the per-manganate, till the faintest red tint is visible. The number of measures required for this purpose—the strength of the per-manganate being known—indicates exactly the quantity of protoxide of iron which the solution contains.

65°. *To estimate the peroxide of iron in the solution* is nearly as easy. It involves, however, two operations.

a. To the acid solution from the soil—which may or may not be prepared in an atmosphere of carbonic acid—add a few small pieces of sheet zinc, cork the flask loosely, and heat to boiling. The zinc dissolves, and the hydrogen given off reduces the peroxide of iron in the solution to

the state of protoxide. If the solution be quite colourless when all the zinc is dissolved, or only of a pale green, the de-oxidation is complete. · If it is still brownish, a little more zinc is added, and the solution again heated to boiling. *No zinc must be left undissolved.* The whole of the iron is now in the state of protoxide.

b. To this solution add that of the per-manganate as before, till the pale red tint becomes visible. From the measure employed, the whole quantity of iron in the soil is easily calculated. And if from this whole quantity we deduct that which is in the state of protoxide as already found (64°), we have the quantity which is present in the state of peroxide.

In making these determinations, greater accuracy is attained by taking a bulk of pure distilled water equal to that of the mixed solutions employed, and ascertaining by experiment how much of the per-manganate solution is necessary to impart to the water a visible red tint. This quantity must be deducted from that which was added to the iron solution, as it was expended in colouring the liquid, and in not per-oxidising the iron.

These methods are much more simple and expeditious than those described in the preceding chapters (34° and 51°), but they are susceptible of great accuracy. In the analysis of a soil, however, we can only employ them as auxiliary processes, and to test our other methods. We cannot add the per-manganate to the acid solutions from which all the other constituents of the soil are to be separated, and therefore, in making a somewhat full and complete analysis of a soil, we must still separate and estimate the iron by the methods previously described.

§ III. ANALYSIS OF IRON ORES.

66°. It is chiefly in the analysis of substances which contain the oxides of iron only, or in which we desire to estimate the quantities and proportions of the two oxides of iron only, that this method becomes of immediate practical value.

a. We prepare our standard solution of per-manganate, or test it anew for the occasion.

b. We dissolve our iron ore—say 100 grains—in muriatic acid by the aid of heat; dilute, filter, and divide into two equal portions.

c. Into the one we pour the solution of per-manganate till the visible tint appears, and from the measure used we calculate the quantity of protoxide of iron.

d. Into the other, contained in a flask, we introduce a few pieces of zinc, dissolve completely by the aid of heat—satisfying ourselves, by the disappearance of colour, that the whole of the iron has been changed into protoxide. We then add per-manganate of potash again, and determine the whole iron which the quantity of ore employed (100 grains) contains.

The larger weight (from *d*) indicates the total per-centage of iron which is contained in the ore; the smaller number (from *c*), the per-centage in the state of protoxide; and the smaller, deducted from the larger, gives the per-centage in the state of peroxide.

In iron ores the protoxide of iron very frequently exists in the state of carbonate. It is so, for the most part, in the clay iron ores, in the oolite ironstone beds, and in many

others. In such ores, every 100 grains of iron in the state of protoxide are equal to 126 grains of carbonate of iron.*

67°. Where it is not desired to determine the proportions of the other ingredients of a clay or other mixed iron ore, the above method is easy, ready, and, in the hands of a good and careful manipulator, very accurate. It is sometimes desirable, however, for economical purposes, to ascertain the proportions both of lime and of clay which an iron ore contains, and even the proportion of phosphoric acid. In such cases the ore must be dissolved in muriatic acid with the aid of heat, and the solution treated in the same way as if it were the acid solution yielded by a soil. And if the insoluble matter of the iron ore is also to be analysed, it must be heated with concentrated sulphuric acid, or fused with the mixed carbonates of potash and soda, as if it were the insoluble matter of a soil, and subsequently treated exactly in the same way (41° to 56°).

Such an examination of course involves both time and labour.

* Thus, of pure metallic iron—

100	grains are equal to	128.53	of protoxide of iron.
100	... ...	142.79	of peroxide of iron.
100	... ...	162.85	of carbonate of iron; and
100	... of protoxide to	126.70	of carbonate of iron.

CHAPTER VII.

GENERAL REMARKS ON THE ANALYSIS OF SOILS.

The organic matter, what it practically suggests.—Soluble saline matters; quantity of, in fertile soils.—In what cases it is necessary minutely to examine them.—Examples of Indian soils.—Soil from the plains of Attica.—How saline matter is to be removed from a soil.—Matters soluble in muriatic acid; which of them may be neglected.—Insoluble matter not always necessary to be analysed.—How to interpret the results of an analysis.

68°. *The Organic Matter.*—The estimation of the total quantity of organic matter in a soil throws light on the two practical questions—how much lime would it be safe to add to it? and in what condition ought we to add it? Where the proportion of organic matter is large, large doses of lime may be applied, and it may be in the caustic state. Where the proportion is small, only small doses of caustic lime are usually admissible, though of unburned lime, chalk, or rich calcareous marl, large applications may be made.

Then, in regard to the organic acids, they are generally the cause of what is called the sourness of a soil. This sourness is removed by quicklime and by wood ashes, so that the use of these is indicated when solutions of carbonate of soda extract much humic and ulmic acids from the soil. Quicklime applied before rain soonest sweetens

the land, though chalk, or chalk marl, or shell sand, perform the same office somewhat more slowly.

69°. *The soluble saline matter* in the soil is rarely very considerable in quantity, and in most cases it will be sufficient to examine *qualitatively* the watery solution of a soil. Cases do occur, however—especially in low flat plains which lie near mountain ranges, or the soil of which is rich in lime—where much saline matter exists in the soil, and is extracted from it by water. Such are some of the soils in India, which, by washing, yield from 1 to 7 per cent of saline matter. Thus in several Indian soils examined by the late Mr. Fleming of Barochan, there were contained in 100 parts—

	1°	2°	3°	4°	5°
Carbonate of lime, . .	7¼	5¼	4¼	4	2
Carbonate of magnesia, .	3¼	2	¼	1	–
Saline matter (chlorides, sulphates, and nitrates,) . . .	1	1¼	3¼	3	7

No. 1 was near Gya in South Behar. Never lies fallow, is covered with water during part of the rainy season —produces from 30 to 50 bushels of wheat per acre.

No. 2. Same district. Not inundated by the rains—produces wheat, pease, cotton, or poppy in the dry, and Indian corn and millet in the wet season. Sometimes manured with wood ashes and cow-dung.

No. 3. From North Behar, Tirhoot. Deep loam, yielding two crops yearly. Not flooded—25 to 30 bushels of wheat per acre.

No. 4. Tirhoot. Light-coloured soil, not so productive as No. 3. Saline efflorescence in patches.

No. 5. Tirhoot. Still less productive; nearly sterile in places from saline efflorescence, except in the rainy season, when it produces good crops of Indian corn.

From these examples we see that from 3 to 4 per cent of saline matter may exist in a soil in certain circumstances, without rendering it unproductive. More than this, however, few soils can contain, and yet continue productive. Where such large quanties occur, the saline matter ought to be washed out and carefully analysed. A large proportion, where the soil continues fruitful, will usually prove to consist of the nitrates of potash, soda or lime. In this country as little as 1 per cent of common salt has been found to prevent crops from growing healthily upon them. The fertile alluvium of the delta of Egypt, contains, in many places, upwards of one per cent of soluble saline matter, but it is not all common salt.

The nature of the saline effloresence which forms upon a soil is always more or less influenced by the nature of the adjoining rocks. Around Durham, where we are in the neighborhood of magnesian limestone rocks, sulphate of magnesia is the prevailing incrustation which forms on the soil in hot dry seasons. In some red sandstone countries, gypsum is a frequent incrustation, and common salt in others; while at the foot of granitic and similar rocks, alkaline salts of various kinds chiefly appear.

The practical cure for soils made barren by excess of saline matter, is the establishment of a thorough drainage. In showery countries these drains will carry away the excess of saline matter which the rains will dissolve and convey to them. In arid countries, the drain must be aided either by artificial irrigation or by yearly natural inundations, such as those to which the salty soils of Tirhoot owe their half-yearly fertility.

70°. I once received a sample of soil from the plains of Attica, with the information that wheat sown at the close of the rainy season ripened and yielded well, but that saline matter rose to the surface in such abundance as gradually to destroy, or entirely burn up the more tender grass; and I was asked to say how grass might be made to grow.

The soil, on analysis, was found to consist of—

Carbonate of lime, ...	38.08
Carbonate of Magnesia, ...	0.73
Sulphate of lime, (gypsum,) . . .	0.18
Phosphate of lime, ...	0,033
Oxide of iron, ...	2.91
Alumina soluble in muriatic acid, . .	2.35
Organic matter, ...	5.75
Salts soluble in water (common salt and sulphate of soda,)	0.20
Insoluble silicious matter (not further analysed,)	50.33
	100.563

This soil was peculiarly rich in lime, but did not overabound in saline matter. It contained all the elements of a fertile soil, and its history evidently showed that it was the saline matter rising with the water from beneath, and left on the surface as the sun of the dry season licked the water up, which was the cause of the agricultural evil. The remedy was easy. "Open drains, that the water which comes from the mountains in the rainy season may carry the saline matter to the sea, instead of lodging in and soaking the soil of the plains, ready to parch the sprouting grass as the dry season progresses." And a similar remedy will cure all similar cases.

71°. *Matters soluble in muriatic acid.*—From open, oamy, or sandy soils, it will seldom happen that, after

boiling in water, any appreciable quantity of potash or soda will be extracted by muriatic acid. The analysis of the acid solution of these soils may therefore be simplified by neglecting the estimation of the trace of alkaline matter they may contain.

Stiff clays, however, are more rich in potash and soda, in states of combination which render them insoluble in water. In the analysis of clay soils, therefore, it will always be proper to examine qualitatively a portion of the acid solution, and thus to ascertain if an appreciable quantity of alkali be present, before deciding upon the exact steps to be taken in conducting the quantitative analysis.

72°. *As to the matter insoluble in muriatic acid*, an ultimate analysis of it is only necessary in very particular instances.

a. That which water takes up represents the actually soluble matter of the soil which is ready *at the time* to minister to the growth of plants.

b. That which is taken up by muriatic acid contains those constituents of the soil which are likely to become available to the plant next in order, as the carbonic and other acids formed naturally in the soil continue to act upon them.

c. That which the muriatic acid leaves undissolved may contain substances valuable to plants, but they are in such a state of combination as only after a long time—or after some energetic chemical treatment, such as the application of quicklime or sulphuric acid—to become available to their use.

In most cases, therefore, the immediately and practi-

cally valuable constituents of the soil may be judged of by a quantitative examination of the soluble parts of the soil only.

73°. Lastly, I would remark that it is only by bringing a very considerable familiarity with practical agriculture to bear upon the results of the chemical analysis of a soil, that correct and practically useful deductions can be drawn from them. In every case, therefore, before finally interpreting these results, we ought to ascertain—

First, The condition of the land as to drainage.

Second, The kind of cropping and manuring to which it has been subjected during the preceeding ten years.

Third, The peculiarities of climate, if any, to which the soil or locality is exposed.

With the aid of these practical elements, if he understand their practical bearing, good economical suggestions may often be drawn by the agricultural chemist from an accurate determination of the chemical composition of a soil.

CHAPTER VIII.

ANALYSIS OF LIMESTONES AND MARLS.

Estimation of the carbonate of lime, and of the carbonate of magnesia.—Complete analysis of a limestone.—Analysis of calcareous marls.

§ 1. ANALYSIS OF LIMESTONES.

74°. *Estimation of the carbonate of lime.*—In many cases it is sufficient for all economical purposes to determine the proportion of carbonate of lime which a limestone contains. This is very easily done.

a. The limestone is dissolved in dilute muriatic acid, hastened, if necessary, by a gentle heat. The insoluble matter is collected on a filter, washed, heated to redness, and weighed. The weight indicates the proportion of insoluble earthy matter which the limestone contains, and thus at once shows whether it is worthy of any further examination. If so—

b. To the filtered solution caustic ammonia is added, and the precipitate collected and weighed as in *a*. This gives the proportion of soluble alumina, oxide of iron, and phosphoric acid, if any, which the limestone contains.

c. To the filtered ammoniacal solution oxalate of ammonia is added; the oxalate of lime collected, washed,

burned, and weighed, as directed as in 33°. The weight represents that of the carbonate of lime contained in the portion of limestone employed.

If the three weights obtained from these three operations, when added together, make up very nearly that of the limestone employed, it may be inferred that little else is contained in it, and the quality of the limestone judged of accordingly.

75°. *Estimation of the carbonate of magnesia.*—But the deficiency may be considerable, or the limestone may be suspected to contain magnesia, the proportion of which it is desirable to ascertain. In this case—

To the solution filtered from the oxalate of lime we add phosphate of soda as long as a precipitate falls, and collect it as described in 37° *b*. Of the phosphate of magnesia obtained, every hundred grains represent seventy-nine grains of carbonate of magnesia in the limstone.*

76°. *Complete analysis of a limestone.*—For most limestones the above examination is sufficient. But it is sometimes desirable to ascertain the quantity of alkaline matter it contains—the phosphoric acid, if any—the soluble silica—and for hydraulic purposes, the composition of the matters insoluble in muriatic acid. With a view to such a complete analysis the following are the steps :—

a. Dissolve in dilute muriatic acid, and collect the insoluble matter.

* The phosphate of magnesia obtained by this process, and the carbonate, are composed respectively of—

	Carbonate.	Per cent.		Phosphate.	Per cent
Magnesia,	20	47.62	Magnesia,	40	35.84
Carbonic acid,	22	52.38	Phosphoric acid,	71.4	64.06
	42	100		111.4	100

b. Boil this insoluble matter in a solution of carbonate of soda, collect on a filter, wash well, and heat to redness.

c. Treat this insoluble matter as if it were the insoluble matter of a soil. Heat it with sulphuric acid, or fuse it with the mixed carbonates of potash and soda, and proceed to separate its constituents as described in 53° to 57°.

d. Add muriatic acid to the soda solution *b* till it is distinctly acid. Evaporate to dryness on the water-bath, stirring towards the end of the process. Drench with muriatic acid, treat with water, and filter. What remains on the filter is silica, soluble in carbonate of soda. It is washed, dried, heated to redness, and weighed.

e. The muriatic acid solution *a* is evaporated to dryness and treated as in *d*. What remains on the filter in this case is silica, soluble in muriatic acid.

f. The acid solution from *e* is treated with ammonia in excess, and the precipitate, if any, is collected and weighed. It contains alumina, oxide of iron, and phosphoric acid, if present, with perhaps a little lime and magnesia.

g. If the precipitate, while still moist, dissolves wholly in acetic acid, it contains no appreciable quantity of phosphoric acid. The iron and alumina are therefore separated by caustic potash in the usual way (34°, 35°).

h. If it is not wholly dissolved by acetic acid, or if, for other reasons, a further research is thought necessary, a separate portion of 500 grains of the limestone is dissolved in muriatic acid, and the solution treated with ammonia in excess. A larger precipitate is thus obtained, containing a more appreciable quantity of phosphoric acid. This precipitate is dissolved in nitric acid, and the phosphoric

acid separated by molybdate of ammonia, and estimated as described in 28° and 40°. Every 100 of phosphoric acid obtained indicate the presence of 217.6 grains of phosphate of lime in the limestone.*

i. The ammoniacal solution from *f* is treated first with oxalate of ammonia to separate the lime, and then with phosphate of soda to separate the magnesia, as already described (37° *b*).

k. Potash and soda will sometimes be found in a limestone; but it will very rarely, indeed, be advisable to seek for it in the residual solution *i*. By reducing a portion of the limestone to *exceedingly* fine powder, and boiling with distilled water, any common salt, or other soluble saline matter which it may contain, will be extracted. By evaporating the solution to dryness, the weight of this may be estimated, and it may, if necessary, be afterwards qualitatively examined, like the watery extract of a soil (19° to 29°).

§ II. ANALYSIS OF MARLS.

77°. By a marl, or a calcareous marl, is generally understood a clay or sand, which contains intermixed with it a variable proportion of the carbonates of lime and magnesia, and often a trace of phosphate of lime. The mode of examining them is nearly the same as in the case of an impure limestone.

* The phosphate of lime consists of—

		Per cent.
Lime, . . .	84.0	54.05
Phosphoric acid, . .	71.4	45.95
	155.4	100.

a. If, on adding muriatic acid, bubbles of gas are given off, it may be inferred that carbonate of lime is present. A weighed quantity is therefore treated with dilute muriatic acid, the insoluble matter collected on the filter, washed, heated to redness, and weighed.

b. To the acid solution ammonia is added, the precipitate collected, washed, and subsequently examined for phosphoric acid, if thought necessary, as in the case of a limestone (76° *h*).

c. From the ammoniacal solution the lime is thrown down by oxalate of ammonia, and the magnesia by phosphate of soda, as already described.

d. If it is thought necessary to examine further the matter insoluble in muriatic acid (*a*), it is treated in the same way as if it were the insoluble matter of a soil (53° to 57°).

CHAPTER IX.

ANALYSIS OF SALINE MANURES.

Saline manures.—Common salt; how it is adulterated and examined.—Nitrate of soda.—Numerous adulterations; how to detect them.—Sulphate of soda; how to test it.—Sulphate of lime, or gypsum.—Sulphate of ammonia and sal-ammoniac; mode of examining them.

78°. THE use of portable manures, either natural or artificial, has now become so very extended that a simple popular method of testing them has become very necessary both to the dealer and to the purchaser. Such simple means I shall endeavor to supply in the present chapter.

The portable manures at present in use consist chiefly of unmixed saline manures, of preparations of bones, and of natural and artificial guanos. I shall consider each of these classes in their order.

The saline manures which are applied to the land in this country in an unmixed state are common salt, nitrate of soda, sulphate of soda, gypsum, and the sulphate and muriate of ammonia (sal-ammoniac.)

§ I. EXAMINATION OF COMMON SALT.

79°. *Common salt* is too low in price to admit of being profitably adulterated with any other saline substance ex-

cept gypsum. But it may be mixed with sand, or it may contain, as natural impurities, traces of chloride of calcium or chloride of magnesium. To detect and estimate these—

a. It should be put into a hot oven in a covered vessel, or dried in some other way, at a heat of 300° Fahr., and the loss of weight noted. This determines the proportion of water it contains, which in moist samples is sometimes considerable.

b. When thus dried, it should be treated with water, and the insoluble matter collected, dried, weighed, and examined. This shows how much earthy or other insoluble impurity it contains, and a slight examination will often be sufficient to show what the impurity consists of. Most of the gypsum, if any is present, will remain in this insoluble matter.

c. Into a pint of boiling water put an unweighed quantity of the salt; stir and add salt till the water is *fully saturated*, and a portion remains undissolved. Let it stand to settle, and then upon a weighed pound of the *dried* salt, reduced to fine powder, pour the clear saturated solution; stir and shake well. Set aside to become clear, and then pour the solution entirely off again. Dry the salt, and weigh it. The loss shows the proportion of the chlorides of calcium and magnesium which the salt contains. This operation is founded on the fact that, after water has been fully saturated with common salt, it is still capable of dissolving these two chlorides, and therefore of extracting them from the common salt with which they may be mixed.

d. If a more minute chemical examination be wished for, the operator will estimate—

The *lime* by oxalate of ammonia (33°). Every 100 grains of the carbonate of lime obtained represent 111 grains of chloride of calcium, or 136 grains of dry sulphate of lime, or 172 grains of common gypsum in the salt.

The *magnesia* by phosphate of soda (37° *b*). Every 100 of the phosphate of magnesia obtained represent 85.3 grains of chloride of magnesium in the salt.

The *sulphuric acid* by chloride of barium (31°). If the same solution be employed for this purpose which has been already used for separating the lime and magnesia, it must be made distinctly sour with pure muriatic acid before the chloride of barium is added.

Every 100 grains of sulphuric acid represent 170 grains of anhydrous or dried gypsum in the salt. From the weight of the lime, as estimated by the previous operation, as much is to be taken as is required to unite with this sulphuric acid to form gypsum. The remainder only of the lime is to be reckoned as chloride of calcium.*

§ II. EXAMINATION OF NITRATE OF SODA.

a. Nitrate of soda is often very moist. A weighed quantity, therefore, should be thoroughly dried in an oven or otherwise at about 300° Fahr., and the loss of weight ascertained. This gives the proportion of water it contains.

b. The dry salt may now be treated with water, and

* Chloride of calcium consists of—

Calcium,	20.
Chlorine,	35.46
	55.46

the insoluble matter, if any, collected, dried, and weighed. This gives the proportion of earthy or other insoluble matter which it may naturally contain, or with which it may have been artificially adulterated. If the earthy matter effervesces when acid is added, it consists in part at least of chalk.

c. Of soluble salts the nitrate may contain, naturally, sulphate of soda, sulphate of magnesia, sulphate of lime, (gypsum), and common salt. These impurities often occur mixed with it in the native nitre-beds, but they may be added also by way of adulteration. For the latter purpose, crystallised carbonate of soda, which is cheaper than the nitrate, may also be added.

d. When common salt is thrown upon red-hot coals, it sparkles or flies about with a crackling noise (decrepitates). Nitrate of soda, and the other salts above named, do not behave in this way. If a decrepitation take place, therefore, when the nitrate of soda is thrown upon red coals, the presence of common salt may be inferred. Its quantity, at least, whether it is great or small, may also be guessed at by the quantity of this decrepitation.

To estimate the exact amount of common salt in the nitrate, however, a weighed quantity must be dissolved in distilled water, and precipitated by nitrate of silver, as described in 32°.

e. If the addition of nitric or muriatic acid, or of vinegar, to a saturated solution of the nitrate, causes bubbles of carbonic acid gas to appear, then it is adulterated with carbonate of soda. The simplest way of determining how much carbonate is present, is to put a solution of a weighed quantity of the nitrate into a stoppered bottle,

to add clear lime water as long as a precipitate of carbonate of lime falls; to cork well up, shake, and allow the white powder to subside. The clear solution is then to be poured off, the bottle filled with boiled distilled water, and allowed again to subside. The clear liquor is again poured off, and the precipitate collected quickly on a filter, dried, heated to dull redness, and weighed. Every 100 grains of this carbonate of lime represent 133 grains of dry, or 358 grains of crystallised carbonate of soda.*

f. To ascertain if it is adulterated with sulphate of soda or sulphate of magnesia, a hot solution of the nitrate is to be made slightly acid with nitric acid, to decompose any carbonate which may be present. If, on the addition of nitrate of baryta, no milkiness appears, it contains no sulphate; but if it become milky, sulphuric acid is present.

If, on the addition of lime-water, a white gelatinous precipitate appear, it contains sulphate of magnesia, if not sulphate of soda; and perhaps a little sulphate of lime may also be present.

To determine the quantities of these ingredients, a weighed portion of the salt is dissolved, and the solution divided into two equal parts.

From the first, sulphuric acid is thrown down by nitrate of baryta (31°).

* Common crystallised carbonate of soda contains no less than 62 per cent of water, its composition being—

Soda, ...	21.8
Carbonic acid, ...	15.4
Water, ...	62.8
	100

—See note, p. 53.

From the second, lime, if present, by oxalate of ammonia (33°), and afterwards magnesia by phosphate of soda (37 *b*).

§ III. EXAMINATION OF CARBONATE OF SODA.

80°. Crystallised carbonate of soda is rarely adulterated in the English market. It may be more or less purely prepared, however, containing traces of common salt and sulphate of soda. If a crystal, when heated to perfect dryness in an oven, loses 62 per cent of water, it may be considered as nearly pure.

It is the dry powdery sodas of the shops which are most subject to adulteration. Dry sulphate of soda and finely-powdered common salt are the mixtures it usually contains. To detect them, a hot concentrated solution of the soda is treated with nitric acid, till all escape of carbonic acid gas ceases. If much sulphate of soda be present, a strong solution of chloride of calcium (prepared by saturating muriatic acid with chalk) will make it milky, or even thick with precipitated sulphate of lime (gypsum). The presence of common salt may be detected by the taste of the dry carbonate, and by its crackling in the fire.

The proportion of sulphate may be determined by nitrate of baryta (31°). Every 100 grains of sulphuric acid indicate 178 grains of dry, or 403 grains of crystallised sulphate of soda.

The proportion of common salt is estimated by means of nitrate of silver (32°).

§ IV. EXAMINATION OF SULPHATE OF SODA.

81°. Crystallised sulphate of soda is rarely adulterated because of its cheapness. It consists of—

		Per cent.
Soda, ...	31.2	19.35
Sulphuric acid. . .	40	24.82
Water, ...	90	55.83
	161.2	100.

so that, if a crystal, when thoroughly dried, loses about 56 per cent of water, it may be looked upon as practically pure.

The dry powdery sulphate of the shops usually contains common salt (which is still cheaper), and some times gypsum. By dissolving in the smallest possible quantity of water, the greater part of the gypsum will remain behind in the state of a white powder, and may be collected. If much common salt be present, it may be detected by the taste. Its exact quantity can only be determined by means of nitrate of silver (32°).

§ V. EXAMINATION OF GYPSUM.

82°. Gypsum is sold either in the unburned or burned state. The unburned, crushed or ground gypsum is rarely adulterated. By washing with water, it is easy to ascertain if there are any earthy uncrystalline substances among it. When heated to redness, this crystallised gypsum loses 21 per cent of water; and by this test its freedom from admixture may also be judged of.

Gypsum in the unburned and burned states consists of—

	Unburned or crystallised.	Burned or dry.
Lime, . . .	28	28
Sulphuric acid, . .	40	40
Water, . . .	18	0
	86	68

Burned gypsum crushes easily, and forms a fine white powder. The only adulterations its price admits of are slaked lime, chalk and pipe-clay. If it give off no bubbles of gas when first moistened, and then treated with an acid it contains no chalk. If, when mixed and shaken up with water, it give a clear solution which is *not* rendered milky by blowing air through it from the lungs for a length of time, it contains no quicklime. And if, when heated with sulphuric acid, and then treated with water, it give a solution from which ammonia throws down no alumina, it is not adulterated with clay (see 54°)

§ VI. EXAMINATION OF SAL-AMMONIAC AND SULPHATE OF AMMONIA.

83°. These salts, when pure, are colourless—dissolve easily and without residue in cold water—when mixed with slaked lime, give off a strong smell of ammonia—and when heated over the lamp, entirely volatilise. They should therefore—

First, be mixed with slaked lime, to ascertain if they really contain ammonia.

Second, be treated with water, and the proportion of insoluble matter, if any, estimated.

Third, heated to incipient redness, till all vapours

and smell cease, and the residue, if any, then weighed and examined.

If the salt begin to rise in vapours when heated to 300° or 350° Fahr., and gradually disappear without melting, it is sal-ammoniac; but if it melt below 300° Fahr. (284), and do not begin to rise in vapour below 536° Fahr., it is sulphate of ammonia.

By these tests the two salts of ammonia can readily be distinguished. If anything remains unsublimed on heating to dull redness, it will be sulphate of soda, common salt, or gypsum. The nature of this residue can readily be recognised by the rules already given.

The two salts of ammonia consist respectively of—

Sal-Ammoniac.		Per cent.	Sulphate of Ammonia.		Per cent.
Ammonia,	17.0 or	31.8	Ammonia,	17 or	25.76
Muriatic acid,	36.4 "	68.2	Sulphuric acid,	40 "	60.61
			Water, .	9 "	13.63
	53.4	100		66	100

So that sal-ammoniac, weight for weight, contains one-fifth more ammonia than the sulphate of ammonia does.

CHAPTER X.

EXAMINATION OF BONE MANURES, GUANOS AND OIL-CAKES.

Crushed bones.—Dissolved bones; how to test them.—Superphosphate of lime; different varieties of, how examined.—Natural guanos; mode of examining—Artificial guanos and mixed manures; analysis of.—Examination of oil-cakes used as manures.

§ I. EXAINATION OF BONES, CRUSHED AND DISSOLVED.

84°. *Crushed bones,* or bone dust, are occasionally adulterated with earthy admixtures. The presence of such adulterations may be detected—

First, By mixing with water, when the lighter bone may be washed off, leaving the heavier sand and earthy matter at the bottom.

Second, By burning a weighed portion in the air at a red heat, and weighing the ash or residue. If the ash exceed half the weight of the bones, earthy or other matter has been added to them.

85°. *Dissolved bones.*—By mixing crushed bones with one-third to one-half their weight of commercial sulphuric acid, they are reduced after a time into a pulpy state. When *dried by admixture with more bone dust,* and laid

in a heap for some time, they form an excellent preparation, which is sold under the name of *dissolved bones*.

The above mode of preparation gives *pure* dissolved bones; but when manufactured for sale it is usual to dry the wet mass by mixing it with gypsum, with saw-dust, charcoal powder, dried peat, ground chalk even, and many other comparatively worthless, if not actually injurious, substances.

Dissolved bones ought to be sour to the taste, and water mixed with and allowed to stand upon them should become distinctly sour. When stirred with water, and the lighter parts poured off, they ought to leave no sand or other heavy earthy matter behind. The presence of saw dust, charcoal, peat, and other vegetable matters, may be ascertained by examining the lighter portions which are washed off.

86°. For a more minute examination the following steps may be taken:—

a. Heat a weighed portion to 350° Fahr. as long as it loses weight. The loss is water. Heat now to redness in the air till everything combustible is burned away, and weigh again. The second loss consists of vegetable matter, if any is present, of the gelatine of the bones, and of the excess of sulphuric acid.

b. Digest the burned residue in dilute muriatic acid to dissolve the phosphates; filter, wash, heat to redness, and weigh the undissolved portion. It consists of gypsum and other earthy impurities which the manure may contain.

c. Boil this insoluble matter in a solution of carbonate of soda, collect on the filter again, and treat with dilute muriatic acid. If it entirely dissolves with effervescence, the insoluble matter from *b* has been gypsum only. The

boiling with carbonate of soda has converted it into carbonate of lime. If it does not dissolve in dilute muriatic acid with effervesence, it consists of clay or other earthy impurities which have been added to the manure.

d. To the acid solution from *b* add ammonia in excess. Collect the precipitate, wash, dry, heat to redness, and weigh. It consists of phosphate of lime, with a little phosphate of magnesia. Every 100 grains of this precipitate indicate the presence of about 200 grains of bones or bone-dust in the manure. This is the really valuable ingredient in the manure; and if it has been prepared from bones at all, its worth may be calculated from the quantity of bones indicated by the weight of the mixed phosphates thus obtained.

87°. The weight of sulphuric acid added to the bones by the manufacturer is not usually quite enough entirely to decompose and render them soluble. But as the immediate efficacy of prepared bones as a manure is very much determined by the proportion of them which is so rendered soluble, it is desirable to ascertain how far this effect has been produced in the sample we are examining. For this purpose—

a. A weighed quantity is to be digested with the aid of heat in a large bulk of water—say 500 grains in a pint of water—frequently stirred, allowed to cool, &c., and finally filtered.

b. Ammonia is added in excess, the phosphate of lime which is thrown down collected on a filter, washed, heated to redness, and weighed. Every 100 grains indicate 200 of bones contained in the manure in a soluble state.

c. To the filtered solution, ammoniacal sulphate of

magnesia is added as long as a precipitate falls (40*c*°). The precipitate, after twelve hours, is collected, dried, heated to redness, and weighed. Every 100 grains of the phosphate of magnesia obtained indicate the presence of about 300 grains of bones in the manure in a soluble state.

The two weights of bones indicated by *b* and *c* are added together, and the sum subtracted from that of the whole weight of bones in the manure (87°*d*). The remainder is the proportion of bone-dust in the manure which is not in a soluble or immediately available state.

§ II. EXAMINATION OF SUPER-PHOSPHATE OF LIME.

88°. By super-phosphate of lime was meant at first simply burned bones rendered soluble by means of sulphuric acid. This is easily examined.

a. A weighed portion is boiled in water for some time, thrown on a filter, and washed. The solution contains the soluble phosphates, which may be separated and estimated, as in 86°, *d*.

b. The insoluble matter on the filter is now boiled for an hour in a solution of carbonate of soda. The sulphate of lime, by this process, is changed into carbonate of lime. This is collected on a filter, washed, heated to redness, and weighed. If pure, or unmixed with sulphate and phosphate, this carbonate of lime will dissolve with effervescence in dilute muriatic acid, and will not be precipitated again by caustic ammonia in excess. Every 100 grains are equal to, or contain the same quantity of lime as, about 200 grains of ordinary bone dust,* or as 115 grains of bone-ash.

* Or as about 170 grains of clean, perfectly dry bones.

c. But if ammonia throws down a precipitate from the solution of this carbonate in muriatic acid, it is phosphate of lime, which remained insoluble in water *a*. It is to be thrown down and collected, as in 86° *d*.

The soluble phosphate from *a*, added to the insoluble phosphate from *b*, gives the whole quantity of phosphate contained in the portion of manure examined.

89°. But by way of improving this variety of super-phosphate, bones themselves instead of bone-ash were afterwards dissolved by means of sulphuric acid, and this preparation was and is still sold by many under the name of super-phosphate. The mode of examining this variety has been explained in the preceding section.

90°. Now, however, the commercial super-phosphates are manufactured by dissolving bones or bone-ash with a variable proportion of powdered mineral phosphate of lime, and mixing the whole intimately together. This variety is not so valuable as either of the others above mentioned, chiefly because the mineral phosphates employed contain always a large quantity of carbonate of lime and of insoluble earthy matter. Upon this lime much of the sulphuric acid is wasted in converting it into gypsum, and the manure is contaminated with an uncertain proportion of comparatively useless ingredients. The samples brought into the market, however, differ very much in value, and it may often be desirable to test and compare them.

A tolerable approximation to the value of a sample may be made by boiling a weighed quantity, as described under 88°, and calculating the proportion of dry bone-earth —the chief valuable ingredient which the manure contains.

For a more detailed examination, the method given for examining dissolved bones (86°) may be employed.

§ III. EXAMINATION OF NATURAL GUANOS.

91°. Of simple tests which can readily be applied to a natural guano, the three following are within the reach of every one:—

a. Dry a weighed quantity at 212° Fahr. The loss of weight is water, and shows whether it has been unduly moist. This operation can be performed by placing a thin basin upon a pan of boiling water, spreading the guano on the bottom of the basin, and covering it with a paper till it has become dry.

b. Mix the guano with slaked lime. If a strong smell of ammonia is given off, it may be presumed to be rich in this valuable ingredient.

c. Mix a quantity of the guano with water, and stir well; allow it to settle for a few minutes, and pour off the lighter matters which still float on the water. Repeat this till all the lighter part is washed away. It will now be seen if the guano is mixed with much sand or gravel. And if a weighed portion of guano has been thus washed, the sandy matter which remains can be dried and weighed, and its proportion determined.

92°. A more thorough analysis of the guano is to be performed as follows:—

a. It is to be dried at 212° Fahr., and the water determined as before. From strong-smelling guanos a little ammonia is driven off by this drying, but it rarely amounts to 1 per cent, and can be allowed for if necessary.

b. The dried guano is to be heated to redness in the air, till everything combustible disappears. The loss consists of organic matters and ammoniacal salts.

c. The burned residue is to be treated with dilute muriatic acid, till everything soluble is taken up. The insoluble matter collected on the filter, washed, heated to redness, and weighed, gives the proportion of useless gravel, sand, silica, or other matters which the guano contains.

Or this burned residue may be washed first with distilled water, to dissolve out the saline matter (*g*), and then treated with acid as here described.

d. Among these other matters, gypsum, added as an adulteration, may be one. If this be suspected, the insoluble matter is boiled in a solution of carbonate of soda, again collected on a filter, and washed. If it now dissolves with effervescence in muriatic acid, it has been gypsum; if only part of it dissolves, the insoluble part can be washed, dried, and weighed, and the gypsum estimated from the loss.

Or, if greater accuracy be desired, ammonia may be added to this solution (*d*) in muriatic acid, and the lime afterwards precipitated by oxalate of ammonia, collected, and heated to redness (33°). Every 100 grains of the carbonate of lime obtained indicate 136 grains of burned gypsum in the insoluble matter from *c*, or 172 grains of common gypsum in the manure.* This method is more accurate, because the carbonate of soda may have dissolved some silica from the insoluble matter, which would propor-

* In the manure the gypsum will exist as common gypsum, containing 21 per cent of water; but as obtained from *c* it is burned gypsum, having been heated to redness.

tionately increase the apparent proportion of gypsum if calculated from the loss.

e. To the acid solution from *c*, ammonia is added in excess. The phosphate of lime of the guano, generally mixed with a little phosphate of magnesia, falls. This is collected on a filter, washed, heated to redness, and weighed.

f. If carbonate of lime or chalk have been present in the guano, it is contained in the filtered solution from *e*. It is estimated by adding oxalate of ammonia, and collecting and heating to redness the oxalate of lime in the usual manner, (33°).

g. By now evaporating the filtered solution from *f*, and heating to dull redness to drive off the ammoniacal salts, the saline or *alkaline matter* contained in the guano is obtained. Or it may be extracted at once by washing the burned residue from *b* with distilled water, and evaporating the solution to dryness.

It will rarely be necessary to analyse this saline matter further. It generally consists of sulphate of soda and common salt, with a trace of sulphate of potash. But if it be desired to determine the proportions of each, the solution is divided into three equal parts—the sulphuric acid is thrown down from one by chloride of barium(31°), the chlorine from another by nitrate of silver, and the third is treated with caustic baryta, as described in the summary, p. 58, letters *m* and *n*.

h. If it be desired to determine the per-centage of ammonia which the guano contains, a weighed portion is put in a small retort, and covered with a dilute solution of caustic potash. The retort is adapted to a receiver

containing a little dilute muriatic acid, and then, by the aid of heat, the matter in the retort is distilled to dryness. The ammonia which passes over combines with the muriatic acid in the receiver, and by evaporating the whole of the liquid to dryness in the water-bath it is obtained in the state of sal-ammoniac. Every 100 grains of this sal-ammoniac indicate the presence of 31.8 grains of ammonia in the guano. (See also 39° *a*.)

§ IV. EXAMINATION OF ARTIFICIAL GUANOS AND MIXED MANURES.

93°. Artificial guanos have generally dissolved bones or some variety of the so-called super-phosphates for their basis. To this are added variable quantities of common salt, sulphate of soda, sulphate of magnesia, sulphate of ammonia, and, more rarely, nitrate of soda. Useless additions of gypsum, burned clay, ground bricks, ochre, &c., to give the mixture a color, and to lower its price, are also made by many manufacturers of artificial manures.

a. An artificial guano should be subjected to the same general treatment as a natural guano. It may be first tested roughly for water, ammonia, and sandy or earthy admixtures, as described under 91°. If a further examination or analysis be thought necessary, then the water, the organic matter, the sand, clay, and gypsum, the phosphates and the lime, are to be estimated as in the preceding section (92°, *a* to *g*).

b. If the soluble salts obtained by the processes *g* have been found, by a preliminary testing, to contain magnesia, this earth may be separated by caustic baryta, as described

in 37° *e*, and the alkalies afterwards estimated either separately or together, as described in 38°.*

c. The ammoniacal salts are also determined as in a natural guano (92° *h*).

d. The nitrates, if present, would be destroyed by heating the artificial guano to redness. They must, therefore, be extracted with water from the guano, or mixed manure as it comes to market. The filtered watery solution must then be tested for nitric acid, as described in 27°. Then if nitric acid be present, and the quantity of chlorine and sulphuric acid has already been determined, the weight of this acid is calculated from the loss.

§ V. EXAMINATION OF OIL-CAKES.

The cake obtained from many varieties of oily seeds when crushed in the mill, is used in England, and in many other countries, as a manure. It is chiefly rape-cake, hemp-cake, mustard-cake, cotton-cake, and some others, not much relished by cattle, which are so employed.

Linseed, poppy, nut, and other sweet oily cakes, which are relished by cattle, and bring a high price in the market, are frequently adulterated with less costly materials. Those which are used as manures are said to be so adulterated also. Where such admixtures of cheaper substances are suspected, the following steps may be taken:—

1°. Reduce the cake to powder, and burn a weighed portion of it in the open air, till everything combustible burns away. The ash should be nearly white, and should not exceed seven or eight per cent of the weight of the cake.

* See also the summary in page 58, under letters *l*, *m*, and *n*.

2°. Treat the ash with diluted muriatic acid. It should nearly all dissolve, leaving no material quantity of sand behind.

3°. To the filtered solution add ammonia in excess, when phosphate of lime will fall. Collected, dried, and heated to redness, the weight of this phosphate should be equal to three-fifths of the whole weight of the ash. If it is much less, some admixture may be suspected.

4°. Reduced to powder, and treated with ether, a genuine oil-cake will yield from ten to twelve per cent of oil. This may be obtained in a separate state by pouring off the ether into a small open glass vessel, when the ether will evaporate, and leave the oil behind.

5°. By breaking the cake into small pieces, treating with repeated portions of boiling water, and then squeezing the insoluble remainder in a thin linen cloth, the husk of the original seed will be obtained in a separate state. An examination of this residue will show whether any foreign seeds have been mixed with the cake, what they are, and to what extent the adulteration has been carried. It is easy in this way, for example, to detect the husks of the small and lighter coloured seeds of brown, or, as it is commonly called, black mustard.

6°. When mustard-seed is crushed and mixed with cold water, it emits the strong pungent smell for which pure mustard is distinguished. Rape-seed, so crushed and mixed with cold water, exhibits also a sensible pungency of *smell*, greater sometimes than that of mustard-seed, but which varies in strength with the sample of rape-seed examined. In *taste*, however, the mustard-seed is much the stronger.

Mustard-cake and rape-cake, *prepared by cold crushing*, exhibit the same properties as the seeds they are made from. If we suspect an adulteration of rape with mustard cake, therefore, we reduce the suspected mixture to a fine powder and mix it with cold water. We reduce to powder also a sample of pure rape-cake, and mix it with water, by way of comparison. The *smell* of the pure rape will probably be quite equal in pungency to that of the sample adulterated with mustard; but the taste will be more pungent in proportion to the quantity of mustard-cake present.

In this experiment boiling water must not be used, as that at once destroys, not the pungent smell, but the pungent taste, both of rape and of mustard, in the form either of seed or of cake.

The above method of testing applies to cakes prepared by cold crushing; but if, during crushing, either mustard or rape seed be heated above 176° Fah., it no longer gives the characteristic pungent smell, when afterwards powdered and mixed with cold water. Most of the rape-cake brought to market has been prepared by *hot crushing* the seeds, at a temperature often as high as 300° Fah. This variety of cake emits very little of the pungent smell, when crushed and mixed with water. Samples occasionally come into the market, however, which have been crushed in the cold; and as these emit a powerful odour, they have sometimes been unjustly called adulterated, and been said to contain quantities of black mustard. Great caution, therefore, must be exercised in coming to such conclusions.

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